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COLOR REMOVAL FROM KRAFT PULP MILL EFFLUENTS BY MASSIVE LIME TREATMENT

John L. Oswalt, et al

International Paper Company
Springhill, Louisiana

February 1973

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Color Removal from Kraft Pulp Mill Effluents by Massive Lime Treatment

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Prepared for

OFFICE OF RESEARCH AND MONITORING
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ABSTRACT

A demonstration plant was installed and operated to determine effectiveness and feasibility of using *massive lime treatment* (that is, 20,000 ppm lime) to decolor kraft pulp mill effluents. The two most highly colored effluents and mixtures of those treated in the demonstration plant were: (1) the almost black effluent from the caustic extraction stage of pulp bleaching, and (2) the light reddish-brown effluent from the final unbleached pulp washing stage. Objectives of the project were to determine:

- effectiveness of color removal
- design and performance of massive lime system equipment
- effects on normal pulp mill operations
- effects on pulp quality
- operating costs

Impact of the massive lime system on a hypothetical 1000 tons-per-day bleached kraft pulp and paper mill is described. Using all the lime normally available in such a mill would allow massive lime treatment of four million of the mill's twenty-nine million gallons of effluent. Such treatment would remove 72% of the total mill effluent's color, reducing final effluent color to approximately 740 APHA units at an estimated cost of $1.80 per ton of pulp (depreciation, insurance, and taxes included).

This report was submitted in fulfillment of Project Number 12040 DYD under the partial sponsorhip of the Water Quality Office, Environmental Protection Agency.

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SECTION I

CONCLUSIONS

The massive lime treatment demonstration plant was operated at International Paper Company's Springhill, Louisiana, mill from February 15, 1970, until August 14, 1971. Results presented in this report led to the following conclusions:

1. Effectiveness of Massive Lime System
   - Massive lime treatment will remove more than 90% of the color bodies contained in effluent from the bleach caustic extraction stage and the final unbleached pulp washing stage. These two effluents contain 65 to 75% of of the total color load produced in the manufacture of bleached kraft pulp.
   - Using all the lime normally available for a typical bleached pulp mill (that is matching the quantity of massive lime sludge produced in effluent treatment with the mill's ability to utilize the sludge for causticizing cooking liquor), approximately 14% of the mill's total effluent can be treated for color removal. Treatment of the most highly colored effluents, the bleach caustic extraction stage effluent and unbleached decker effluent, will remove 72% of the mill's total color load. Final total effluent color discharged from the mill can be reduced from 2630 to approximately 740 APHA units of color.
   - Another benefit of massive lime treatment is a 20 to 40% reduction in BOD_5 of the effluents treated.
2. Design and Performance of Massive Lime System Equipment
   - Foaming and carryover of solids from the primary clarifier was the most serious problem encountered with the demonstration plant as originally designed. It is imperative that equipment throughout the system be designed to prevent air entrainment and eliminate or control foaming.
   - For effluents having a color of 5000 APHA units or more, sludge settling rate and filter rate vary inversely with the concentration of organic compounds in the effluent to be treated. Therefore, for a given flow rate, the primary clarifier and sludge filter sizes must vary with the concentration of organic compounds in the effluent.
3. Effects on Normal Pulp Mill Operations
   - Using massive lime system sludge in the pulp mill's liquor causticizing operation will dilute the cooking liquor, causing its concentration to be approximately 15% lower. Therefore, the volume of liquors handled throughout the pulp mill processes will increase, and increased capacity in most of the chemical preparation and recovery equipment will be required.
   - Lime kiln fuel requirements increase approximately 6.4% for a given pulp production, and increased kiln capacity is required.

- Organic compounds carried with massive lime sludge into the mill's cooking liquor system intensified foaming problems.
- Buildup of chlorides or other materials caused by use of massive lime sludge for causticizing cooking liquor had no apparent effect in the chemical recovery cycle.

4. Effects on Pulp Quality
   - There were no apparent effects on bleachability of the pulp or on finished product quality.

5. Cost
   - For a typical 1000 tons-per-day mill using the massive lime system to treat four million gallons of effluent per day, costs would be approximately $1.80 per ton of bleached pulp. This cost is based on current typical raw material and utilities cost. It includes the cost of depreciation, insurance, and taxes for the increased capital investment for a new mill's color removal system and expanded capacities elsewhere in the mill necessitated by installation of the massive lime system.

SECTION II

INTRODUCTION

The kraft pulping process, used for approximately 90% of the United States' chemical wood pulp production, produces highly colored liquid effluent. During the 1950's the National Council for Stream Improvement (NCSI) now known as the National Council for Air and Stream Improvement (NCASI), conducted many experiments to develop a color removal process for pulp mill effluents. NCSI's work led to development of a process called "The Massive Lime Treatment for Color Removal from Kraft Waste Pulping Liquors." This process was patented in 1964 and assigned to NCSI (Reference 1). Laboratory work by NCASI, (References 2 and 3), International Paper Company (References 4 and 5), Western Kraft Corporation (Reference 6), and probably others showed the massive lime process removed 90 to 97% of the color bodies and 35 to 50% of the BOD from pulp mill effluent. Pilot plant operations conducted by West Virginia Pulp and Paper Company in 1970 (Reference 7), and by Western Kraft Corporation in 1964 (Reference 6) confirmed the laboratory results.

A larger scale evaluation was needed to: (1) evaluate possible effects of the massive lime process on normal pulp mill operations, (2) evaluate possible effects on pulp and paper quality, (3) obtain reliable design criteria for the system and the equipment, and (4) determine the cost of such treatment. Discussions were held in late 1966 between FWPCA and International Paper Company on the possibility of a demonstration project. Subsequently International Paper Company submitted a proposal to the FWPCA to construct, operate, and evaluate a demonstration color removal plant designed to handle up to 530 gpm of pulp mill effluent. On June 11, 1968, International Paper Company accepted an FWPCA Research and Development Grant to evaluate and demonstrate the massive lime process, and if this method of color removal was successful, to determine the best design criteria for future installations.

The demonstration plant was constructed at International Paper Company's mill at Springhill, Louisiana. The plant started operation in February 1970 and operated until August 14, 1971, except for shutdowns due to equipment breakdowns, natural gas shortages, and equipment alterations.

Primary Sources Of Kraft Pulp Mill Effluent Color

The kraft pulping process solubilizes and removes lignin from wood in a pressure vessel (digester) at approximately 100 psi and 340°F. This process is called "cooking." Lignin reacts with chemicals added to the digester in a cooking liquor (white liquor) which is primarily water, sodium hydroxide, and sodium sulfide. Highly colored lignin and other organic chemical compounds formed during the cooking process contribute a dark reddish-brown color to the unbleached pulp. Much of this type pulp is used in grocery bags, corrugated boxes, and other products where the pulp's color is acceptable. For still other paper products however, the pulp is bleached to various degrees of whiteness.

Practically all lignin and other materials dissolved during the cooking process are recovered and used by the mill as fuel for its boilers. The small portion of these highly colored soluble

materials which are not recovered for use as fuel eventually leave the mill in its liquid effluent. *The main source of colored effluent in a kraft pulp mill producing unbleached pulp is the final stage of pulp washing.* This effluent is called the "unbleached decker effluent" because it is discharged from a machine known as the unbleached pulp decker.

If the pulp is bleached, additional lignin is solubilized and removed from the pulp. Bleaching is usually accomplished in multi-stage processes with most of the remaining lignin removed in the "caustic extraction stage"—normally the second step in the bleaching process. Effluent from the caustic extraction stage is almost black. *This caustic extraction stage effluent is the main source of colored effluent in a kraft pulp mill producing bleached pulp.*

Color compounds in the kraft pulp mill effluent have been identified by Holzer (Reference 8) as sulfur dyes derived from phlobotannins. Investigations by the Empire State Paper Research Institute, as reported in NCASI Technical Bulletin No. 239 (Reference 9), indicate the color is due largely to lignin and its degraded products.

Massive Lime Process

Early work on color removal processes had shown most of the color could be removed by treating the pulp mill effluent with chemicals such as alum and lime. However, a voluminous sludge was produced which would not densify on settling. Efforts to concentrate the sludge using filters, centrifuges, and other methods were unsuccessful.

The significant difference between the massive lime process evaluated in this project and earlier processes is in the *amount* of lime used. In earlier work lime usage was closely related to the stoichiometric requirements whereas the massive lime process utilizes a large excess of lime to produce a heavy and readily settleable sludge.

A flow diagram for the massive lime process is shown in Figure 1. Part of the total effluent to be treated is used for slaking the lime which is to be used. Then, the remaining colored effluent is treated with a heavy dose of the slaked lime (approximately 20,000 ppm lime based on weight of the effluent). The mixture is allowed to react for 5 to 30 minutes. During this period color bodies react with calcium ions. The mixture of lime and liquor then enters the primary clarifier where the slaked lime and the insoluble calcium-organic compounds are separated from the treated effluent. Clarified, decolored effluent is then treated with carbon dioxide to precipitate dissolved calcium compounds as calcium carbonate. This calcium carbonate settles in the carbonator clarifier, is removed, and then combined with sludge from the primary clarifier in the sludge storage tank. Clarified, decolored effluent with most of the calcium compounds removed overflows from the carbonator clarifier.

Sludge having a solids content of 18 to 22% is pumped to a rotary vacuum filter and concentrated to about 50% solids. Filtrate from the vacuum filter is returned to the massive lime treating system because of its high content of dissolved and suspended solids. Sludge at approximately 50% solids is removed from the process and the lime in the sludge is recovered by using the sludge in the mill's causticizing process (process in which the mill produces the white liquor used for cooking).

Figure 1

MASSIVE LIME PROCESS

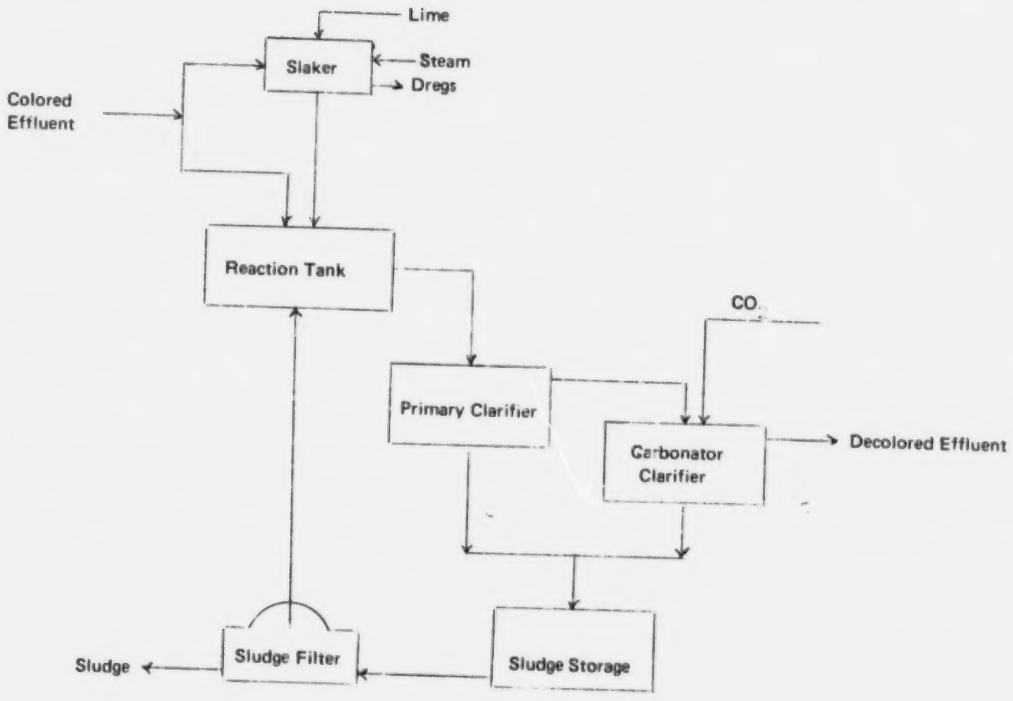


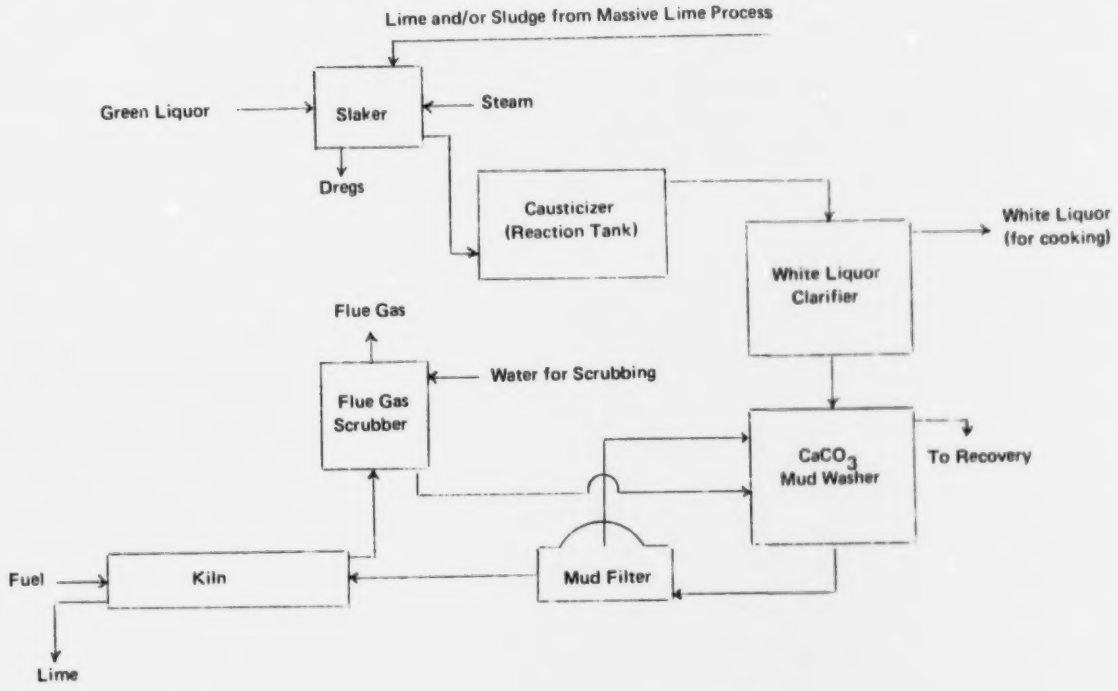
Figure 2 is a flow diagram for the causticizing process. Green liquor which is fed to this process is a water solution of inorganic chemicals, primarily sodium carbonate and sodium sulfide, which have been recovered by burning concentrated spent cooking liquor in a reduction furnace. Sludge from the massive lime process is mixed with green liquor causing reactions which convert sodium carbonate to sodium hydroxide and calcium hydroxide to calcium carbonate. The precipitated calcium carbonate is settled from the mixture and the resulting clarified liquor is the white liquor used for cooking. The calcium carbonate is washed with water to recover soluble sodium salts and is then burned in a lime kiln to convert the calcium carbonate to lime.

Lime produced in the kiln can be recycled to the massive lime process. Carbon dioxide from the lime kiln flue gases can be used for the carbonation step of the massive lime process.

Color bodies removed from the massive lime process with the sludge are to a large extent soluble in caustic. Most of the color compounds dissolve in the white liquor as it is produced in the causticizing process. Therefore, white liquor produced with this process is black. Color compounds which are not dissolved in the white liquor remain with the calcium, go to the lime kiln, and are burned. Color compounds dissolved in the white liquor eventually are burned in the mill's chemical recovery furnace.

Figure 2

CAUSTICIZING PROCESS FOR A KRAFT PULP MILL



SECTION III

DESCRIPTION OF INITIAL DEMONSTRATION PLANT EQUIPMENT

The major manufacturers of equipment for kraft pulp mill caustic plants had little or no experience in the clarification of water using excessive calcium hydroxide or the concentration of the sludge resulting from this clarification. They expressed little confidence in the reliability of such a system, and were reluctant to recommend equipment for the project. Neither International Paper nor the inventors of the massive lime process had any significant experience outside the laboratory in determining type and size of equipment necessary to accomplish the objectives. However, International Paper did have personnel experienced in clarifying pulp mill liquors, and treating water for solids removal.

International Paper Company engineers and the inventors reviewed data from several reports covering laboratory experiments and small pilot plant trials with the massive lime process. Based on these reports, the equipment for this project was decided upon. The demonstration plant was designed to treat 530 gpm of waste water using 20,000 ppm lime (CaO). Figure 3 is a flow diagram showing the original equipment installed. The following is a brief description of the major items of equipment which were installed.

Slaker

Both lime and liquid (colored effluent for the massive lime process) were added continuously into the slaker tank. The lime pellets disintegrate and react with water in the liquid to form calcium hydroxide. A sloping trough equipped with an oscillating rake was used to remove heavy solids (dregs) which settled to the bottom of the slaker. A mixture of liquid and slaked lime overflowed through a weir.

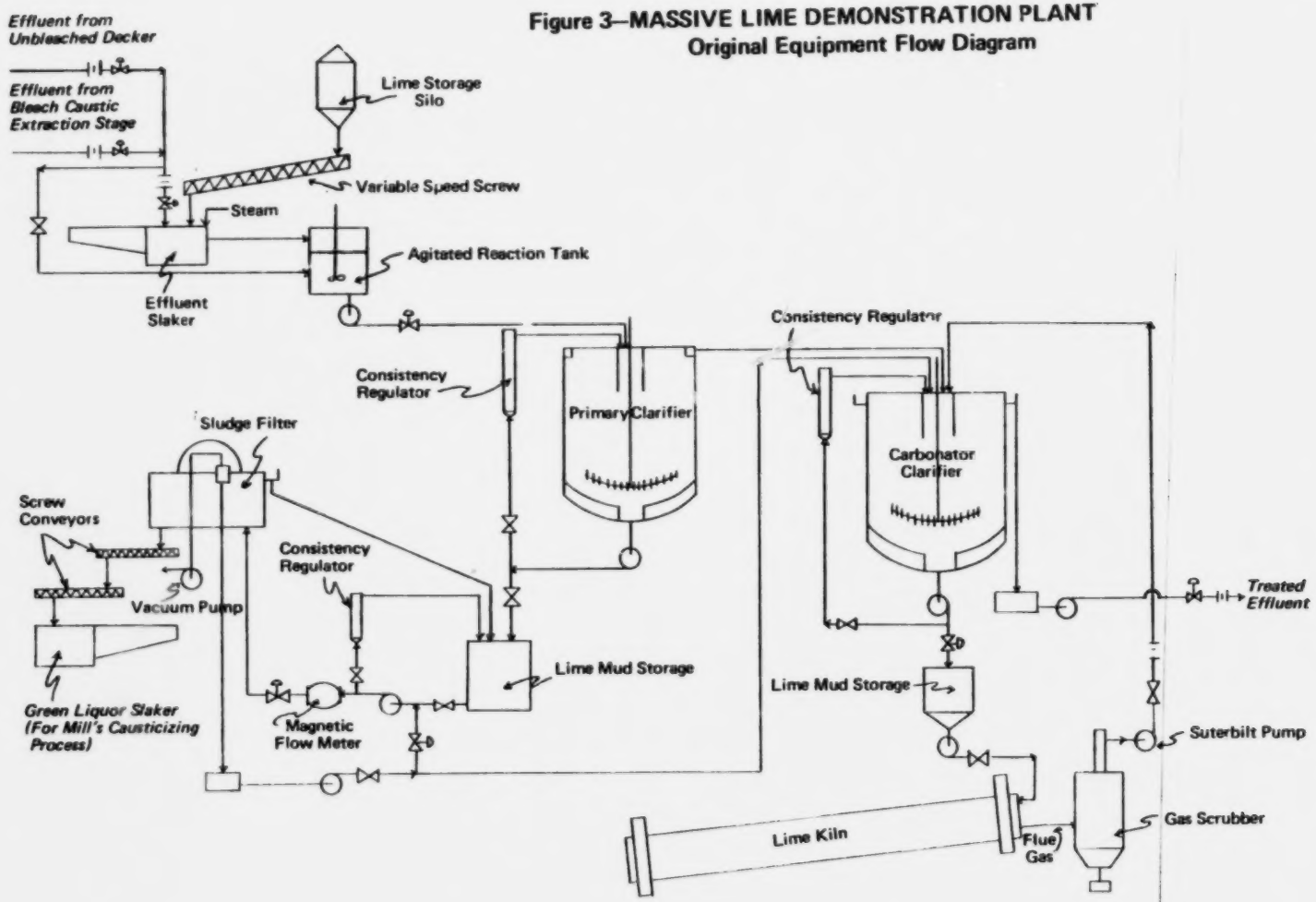
The slaking of lime to calcium hydroxide occurs slowly at normal ambient conditions; however, as temperature is raised the reaction becomes much more rapid. Therefore, temperature was normally controlled to approximately 210°F for slaking.

A typical lime-green liquor type slaker with a capacity for slaking 75 tons of lime per day was installed. This unit was equipped with liquid flow control, temperature control, and a dregs removal rake. Lime feed to the slaker was controlled by a variable speed screw conveyor from the lime storage silo. The two types of effluent to be treated in the demonstration plant (unbleached decker effluent and caustic extraction stage effluent) were piped into the system so that either flow could be treated individually, or a mixture of the two could be treated. Only part of the total effluent flow was needed for slaking, therefore a by-pass was provided so the remainder would go directly to the reaction tank.

Reaction Tank

The agitated reaction tank, 10 feet in diameter and 8 feet high, was equipped with level control operating on the discharge of the pump to the primary clarifier. A minimum of five minutes retention time was provided by the reaction tank.

Figure 3—MASSIVE LIME DEMONSTRATION PLANT
Original Equipment Flow Diagram



In the reaction tank, slaked lime and the effluent to be treated were mixed and reaction occurred between the calcium ions and the organic compounds in the liquid.

Primary Clarifier

From the reaction tank, the slurry is pumped to the primary clarifier, a large tank in which the separation of settled solids and supernatant liquid was accomplished. The clarifier has (1) a center well used as the entrance for the slurry, (2) a rotating rake to convey settled solids to (3) a collection well from which settled solids were removed, and (4) a launder through which the clarified liquid flowed from the tank.

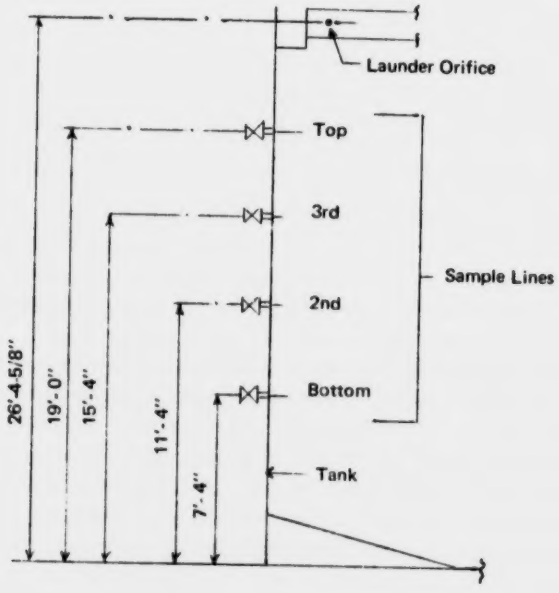
The primary clarifier installed in the demonstration plant was designed for a rise rate of one gallon per square foot per minute, with a retention time of three hours. The tank was 26 feet in diameter and 26 feet high. Clarified liquid overflowed from six three-foot wide weirs, which were equally spaced around the periphery of the tank. A consistency regulator was provided to control the density of sludge discharged from the unit.

When installed, the entrance into the center well was an open ended pipe which discharged above the liquid surface in the center well. The center well was 3 feet in diameter and extended 3 feet below the liquid level in the clarifier.

As shown in Figure 4, four sample lines at different elevations were provided on the side of the clarifier.

Figure 4

PRIMARY CLARIFIER SAMPLE LINES

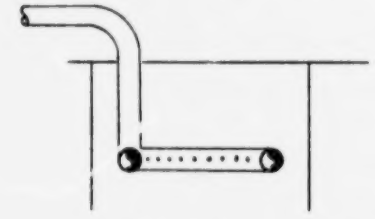


Carbonator Clarifier

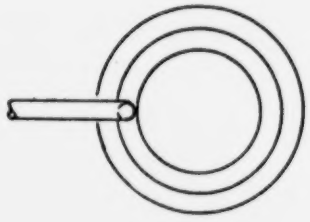
Overflow from the primary clarifier flowed by gravity to the dual purpose carbonator clarifier. It was designed so that carbon dioxide (lime kiln flue gas) was introduced into the clarifier's center well along with the incoming liquid. Originally, the flue gas was added five feet below the surface of the liquid from a circular section of 6-inch diameter pipe having 1/2-inch diameter holes drilled on 4-inch centers (Figure 5). The center well, designed to give a retention time of about 20 minutes, was 15 feet in diameter by 12 feet high with a liquid depth of 10 feet. A loosely fitting cover was provided to minimize the escape of foam created by action of undissolved flue gases.

Figure 5

FLUE GAS DISPERSING ARRANGEMENT IN CARBONATOR



SECTION



PLAN

ORIGINAL DESIGN

Overall dimensions of the carbonator clarifier tank were 30 feet in diameter by 24 feet high. Rise rate between the center well and the outside wall was designed to be one gallon per square foot per minute. Retention time was slightly more than three hours. Clarified liquid overflowed from six three-foot wide weirs equally spaced around the periphery of the tank. Also, as in the primary clarifier, a rotating rake and collection well were provided at the bottom for sludge removal. Sludge from the carbonator clarifier was initially pumped to the mill's existing unfiltered lime mud storage tank (a tank in the mill's normal lime recovery system where a calcium carbonate sludge is collected). A consistency regulator was installed to control density of sludge discharged from the unit.

A positive displacement, constant volume pump capable of supplying 650 cubic feet of flue gas per minute was installed to provide gas for the carbonator.

Sludge Storage Tank

A tank large enough to hold a two-hour surge was installed between the primary clarifier and the sludge filter. This tank's capacity was large enough to allow for backwashing or acid cleaning on the filter, or minor repairs on the filter and causticizing system.

A consistency regulator was provided so that a sludge of uniform consistency could be delivered to the sludge filter.

Sludge Filter

The purpose of the sludge filter was to thicken the mud from approximately 18 to 22% solids to approximately 50% solids. For this a sludge filter of the pre-coat type was installed (similar to existing rotary vacuum drum filters, used as lime mud concentrators for feed to the mill's lime kiln). This filter had an 8-foot diameter by 8-foot wide face. Sizing was based on an assumed capacity of 1000 pounds (dry basis) of sludge per day per square foot of filter area. A Nash pump with vacuum break set at 22 inches was installed to provide the necessary vacuum.

A screw conveyor was installed to transfer filter cake from the sludge filter discharge to the pulp mill's slaker in the cooking liquor causticizing process.

Location

An area near the mill's reburned lime storage silo and near one of the existing lime slakers was cleared and used for erection of the massive lime process slaker, reaction tank, mud filter, and vacuum pump. The filter was located above the new slaker. Another area 150 feet from the silo was cleared for the clarifiers. The sludge storage tank (to receive sludge from the primary clarifier) was located near the filter. A control room was built as an extension to the existing slaker control room.

A small surge tank with an overflow line to the sewer was located on the ground floor of the bleach plant to receive caustic extraction stage effluent from either or both of two bleaching lines. This surge tank was some 2200 feet from the slaker and was serviced by a pump with volume and head characteristics sufficient to deliver the desired flow to the system.

The pump used to supply unbleached decker effluent was tied into the decker seal pit. The decker seal pit was located some 750 feet from the slaker.

Flow indicators and controllers were located in each of the above lines so flow to the system could be controlled independently.

There were two separate systems in the mill's caustic room for slaking, causticizing, clarifying white liquor, and storing white liquor. That part of the system which furnished white liquor

to the bleached pulp mill digesters was isolated for studying effects of using the massive lime system filter cake on the liquor and pulping systems.

Once the demonstration plant started operating, the need for changes in some of the original equipment became obvious. Significant changes were made in some parts of the plant. Problems which made the changes necessary, changes that were made, and a description of the equipment finally used in the demonstration plant are discussed in Section IV.

SECTION IV

OPERATING PROBLEMS AND MODIFICATIONS TO ORIGINAL EQUIPMENT

The massive lime process demonstration plant was completed in January 1970 and turned over to the mill operating department February 1, 1970. The first two weeks of February were used to check out the mechanical and electrical dependability of the equipment, and to check and calibrate instruments and automatic equipment. Also during this period, the people who were to operate this system were trained.

It was immediately apparent that there were serious operating problems with the system as initially installed. Reliable data on performance of the massive lime process could not be obtained until equipment modifications were made to solve these operational problems. The modifications were not completed until January 1971. Major operating problems were:

- foaming and carryover of suspended solids from the primary clarifier
- scaling and mechanical problems with the carbonator system
- poor pH control in the carbonator

Foaming Problems In Primary Clarifier

The demonstration plant started operation on February 15, 1970, treating bleach caustic extraction effluent. Soon after start-up, the surface of the primary clarifier was covered with foam. This foam soon built up enough to overflow into the launders, and at times overflowed the outer wall of the clarifier. It was impossible to observe the liquid overflow from the primary clarifier due to coverage of the entire surface by this tenacious foam. Performance of the clarifier was completely unsatisfactory as long as the foam persisted because of excessive carryover of suspended solids.

Suspended solids (primarily calcium hydroxide) carried over from the primary clarifier varied from 1500 ppm to 7500 ppm. This condition existed regardless of the type of effluent being treated.

The carbonation system did not have enough capacity to carbonate both the soluble calcium hydroxide and the insoluble calcium hydroxide from the primary clarifier. Therefore excessive calcium hydroxide in sludge from the carbonator clarifier caused foaming and operational troubles in the mill's causticizing process mud filter and in the lime kiln.

Foam in the primary clarifier usually had the consistency of shaving cream. Sometimes there were large bubbles, 1/2 to 6 inches in diameter, indicating the presence of entrained air. Commercial defoamers were tried, but their use was costly and not completely effective.

Since the foam was believed to be caused by entrained air, sources of air were eliminated where possible. Leaking packing glands on pumps were repaired. Speed was reduced on agitators which could have been whipping air into the liquid, and splashing of entering liquid was eliminated by submerging entrance piping in all tanks.

These changes eliminated the large bubbles of foam but did not eliminate the very persistent foam having the consistency of shaving cream. It was determined that this foam was caused by minute gas bubbles released from the waste water as temperature was increased in the reaction tank by addition of the slaked lime slurry to the effluent. Speed of the reaction tank agitator was not fast enough to whip air into the liquid, but it was fast enough to keep the minute air bubbles from escaping.

There was nothing practical that could be done to prevent the temperature increase and the resulting release of air bubbles. Therefore, the next approach was to collect the foam and either destroy it or remove it from the clarifier.

A small experimental clarifier was designed, built, and installed adjacent to the reaction tank (Figure 6) and in parallel with the primary clarifier. Thick foam collected and overflowed from the experimental clarifier until a small Yarway shower was installed at the top of the clarifier's center well. The shower was positioned so that its conical spray covered most of the open top of the center well. Liquid used for the spray was the same as the untreated effluent going to the reaction tank. The shower delivered 4 to 5 gpm at 20 psi.

Cylindrical and conical center wells were tried (Figure 6). Both appeared to work, but the conical design may have been slightly more effective. Therefore, when the primary clarifier was modified a conical center well was installed. Overflow from the experimental clarifier was through 8 holes on a horizontal line. These orifices were sized so that liquid level in the tank would be 2 inches above their center line at 18 gpm outflow and 4 inches above their center line at 25 gpm. The orifices discharged into a collection launder and flowed from there to the sewer. This orifice type of overflow launder was essential to allow visual observation of the outflow even if the surface of the clarifier was covered with foam.

Observation of the outlet stream from the orifices showed the effect of many operating variables on the condition of the effluent. Some of the observations were:

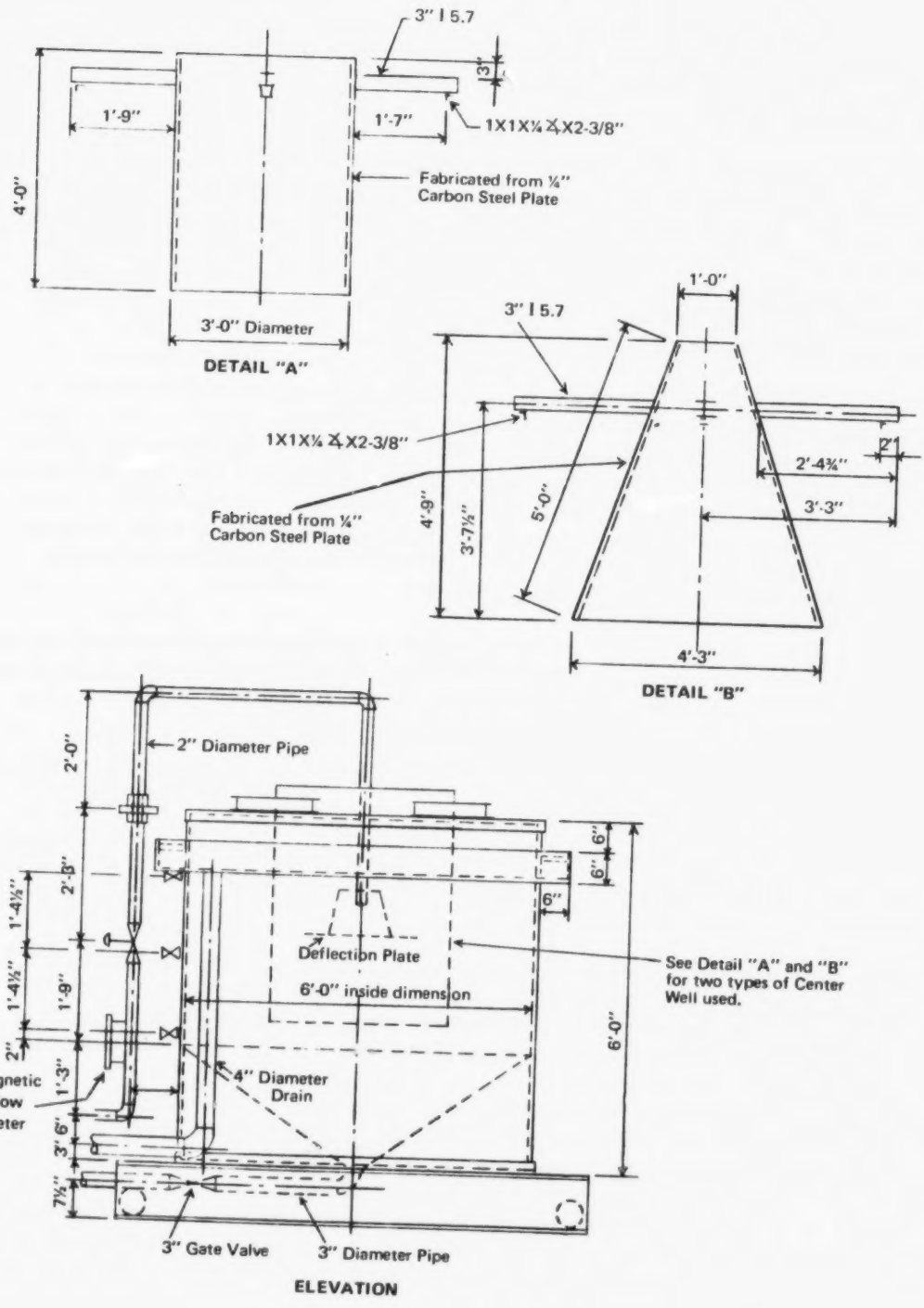
1. When the unit was performing statisfactorily effluent was not turbid.
2. When clarifier's surface was covered with foam, the lower 2/3 of the stream was not turbid, but the upper 1/3 was very turbid from entrainment of the floating foam which contained suspended solids.
3. When the overflow became slightly turbid because of improper operation or overloading the clarifier, conditions should be changed for better operation or concentrated suspended solids would soon overflow. There were conditions under which the top half of the stream would be only slightly turbid and the bottom half would have a heavy concentration of sludge.

When the unit was operating satisfactorily, liquid from the bottom sample port (Figure 4) contained essentially the same percent settled solids as underflow from the pilot unit. Liquid from the second sample port was very turbid, and that from the third sample port was only slightly turbid. If liquid from the second sample port became muddy, mud would soon be overflowing the unit.

During the experiments with the small clarifier, flow to the regular clarifier and carbonator was maintained at 250 gpm (± 10). Operation of the primary clarifier was as disappointing as ever.

Figure 6

EXPERIMENTAL CLARIFIER



High molecular weight polymer settling aids were tried with little or no improvement in settling rate or clarity of the overflow in either the experimental clarifier or the primary clarifier.

Based upon experience gained with the experimental clarifier changes were made in the primary clarifier during the latter part of 1970. Figure 7 illustrates the final configuration of the primary clarifier.

In the experimental clarifier, the velocity of the clarified overflow through the launder orifices had a tendency to pull suspended particles (suspended in the foam) from the liquid surface due to induced eddy currents. To reduce this velocity and the localized conditions that created eddy currents, 6 radial launders from the conical internal to the peripheral launder were added. Even when a light foam or scum covered the surface of the clarifier, the clarity of the overflow could still be observed with the new arrangement. Also, there was very little entrainment from the surface in the outflow.

The most important changes made in the primary clarifier were: installation of the spray showers to control foam, changes in the launders to provide low velocity outflow through orifices instead of overflow from weirs, and installation of the conical center well. These changes (plus the elimination of air which was accomplished earlier) eliminated the foaming and carryover problems of the primary clarifier under normal operating conditions. Foam still rose within the new center well but was destroyed by the sprays; however, whenever level controls in the reaction tank allowed liquid level to drop low enough for air to be pulled into the system foaming was still a problem.

Since essentially three changes in the primary clarifier were made simultaneously, it was not proven which of the changes had the greatest effect. It is possible that with foam controlled through use of the Yarway showers and submerged inlet, the original clarifier design may have been adequate especially at low flow rates (100 to 250 gpm).

Scaling And Mechanical Problems With Carbonator System

Openings in the original carbonation system (Figure 5) rapidly scaled up, limiting the flue gas flow to the carbonator. The holes were enlarged but scale still formed rapidly to restrict gas flow.

After consulting with people who had successfully carbonated waters with high calcium content, design of the carbonator was changed. A header was built above the reaction chamber cover with drops to which spargers were attached. These spargers connected to the header with 1 1/2-inch pipe were inverted cones with scalloped bottoms located 5 feet below the liquid level. Each sparger could be removed without affecting the operation of the others. Figure 8 shows the final sparger design and Figure 5 shows the original arrangement. While the system shown in Figure 8 worked satisfactorily, better equipment and arrangements for distribution of flue gases for carbonating solutions are probably available.

Before modifications were made, mechanical breakdowns and electrical overloads caused the flue gas blower to be out of operation most of the time. The mechanical troubles were primarily rapid deterioration of seal rings, bearings, and associated parts due to the abrasive action of particles in the flue gases.

Figure 7

PRIMARY CLARIFIER INTERNALS

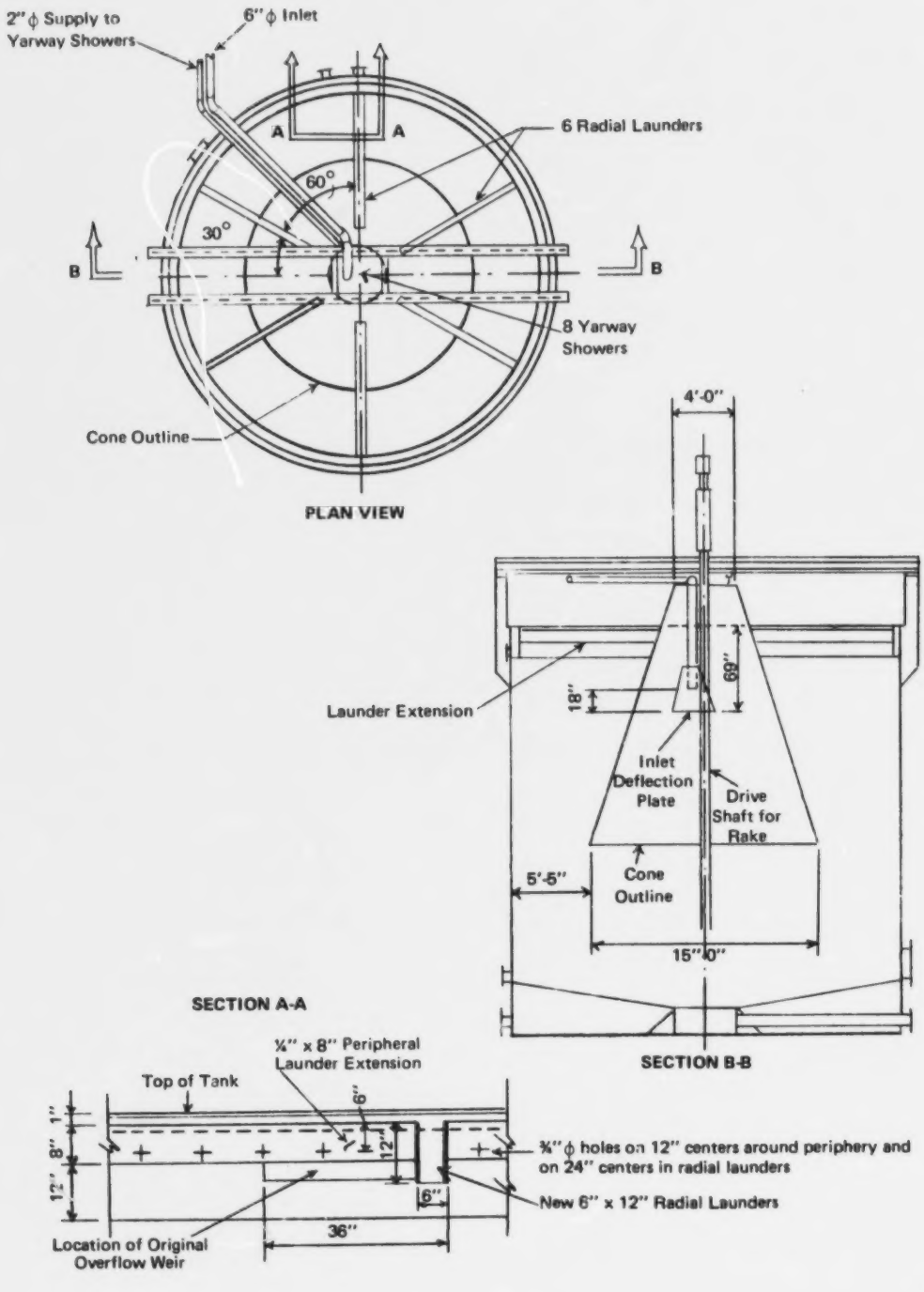
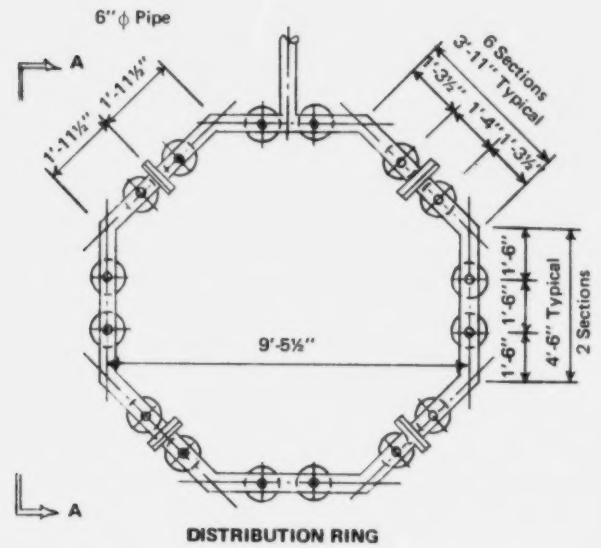
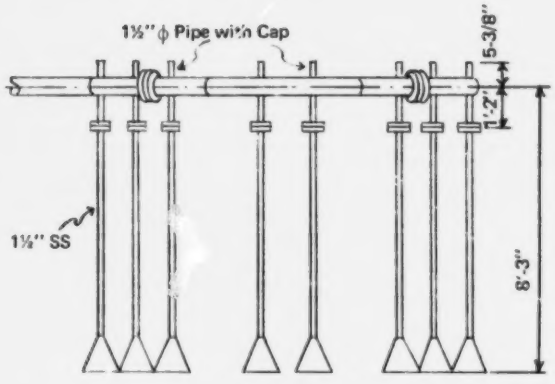


Figure 8

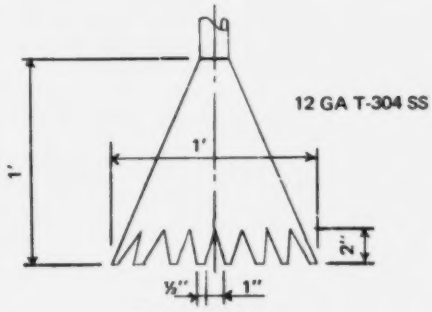
DESIGN OF CARBONATOR SPARGERS



DISTRIBUTION RING



VIEW A-A



SKIRT DETAIL

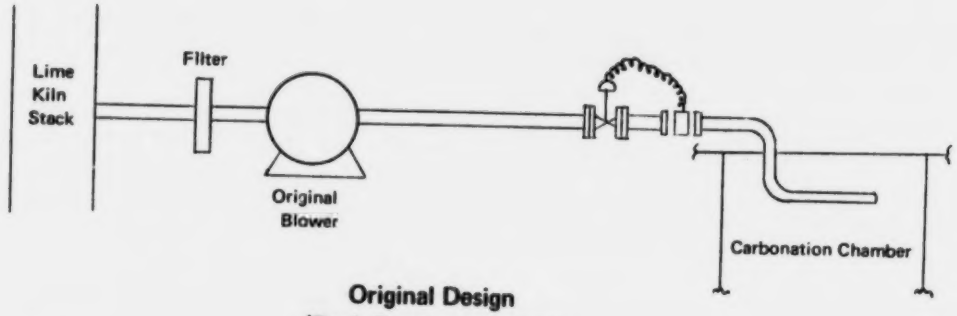
The electrical overloads were attributed to the back pressure type flow control originally installed in the system.

The valve controlling the flue gas flow to the carbonator was changed from a back pressure type control to an atmospheric relief control (Figure 9). The back pressure control would overload the blower, reduce flow and cause condensation of water vapor which led to some of the maintenance problems with the blower. Since a pressure only slightly above the hydraulic depth of the spargers would supply sufficient flue gas for carbonation, atmospheric relief control was better suited to the system. The change eliminated the overload problem, but did not reduce the mechanical breakdowns.

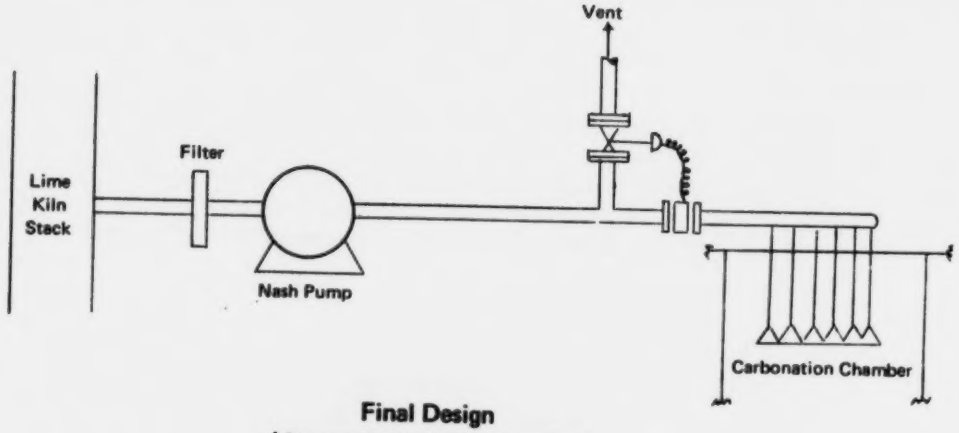
The mechanical breakdowns were attributed primarily to use of a blower not suited for this type application. Therefore the original positive displacement flue gas blower was replaced by a converted Nash vacuum pump. There were no electrical or mechanical problems with the Nash pump during its 8-month period of operation.

Figure 9

FLUE GAS CONTROL



Original Design
(Back Pressure Control)



Final Design
(Atmospheric Relief Control)

Poor pH Control In The Carbonator

The carbonator clarifier pH control as originally installed was unsatisfactory. The pH sensor was located in the clarifier's overflow launder indicating pH some 3 hours or more after carbonation. To correct this fault, a line was run on a 45° angle from inside the reaction chamber to a sample jug outside the clarifier where the pH sensor was relocated. This arrangement gave better pH control.

Other Changes

In addition to the equipment changes which have already been discussed, several other changes were made. The pipe for conveying liquid from the primary clarifier overflow to the carbonator clarifier was replaced with an open trough. This was done to allow easier cleaning since the pipe scaled rapidly, restricting flow to the carbonator clarifier.

Mud from the carbonator clarifier originally was put into the mill's unfiltered mud tank. Since this gave problems in operation of the mill's regular mud processing system, the carbonator clarifier mud was put into the massive lime system sludge storage tank. This resulted in somewhat improved dewatering on the sludge filter. The original system was used only during the period when there was excessive carryover of calcium hydroxide from the primary clarifier. Therefore, inadequate capacity for carbonating in the carbonator clarifier and the resulting presence of calcium hydroxide in the carbonator clarifier mud may have contributed to the problems encountered with the original system.

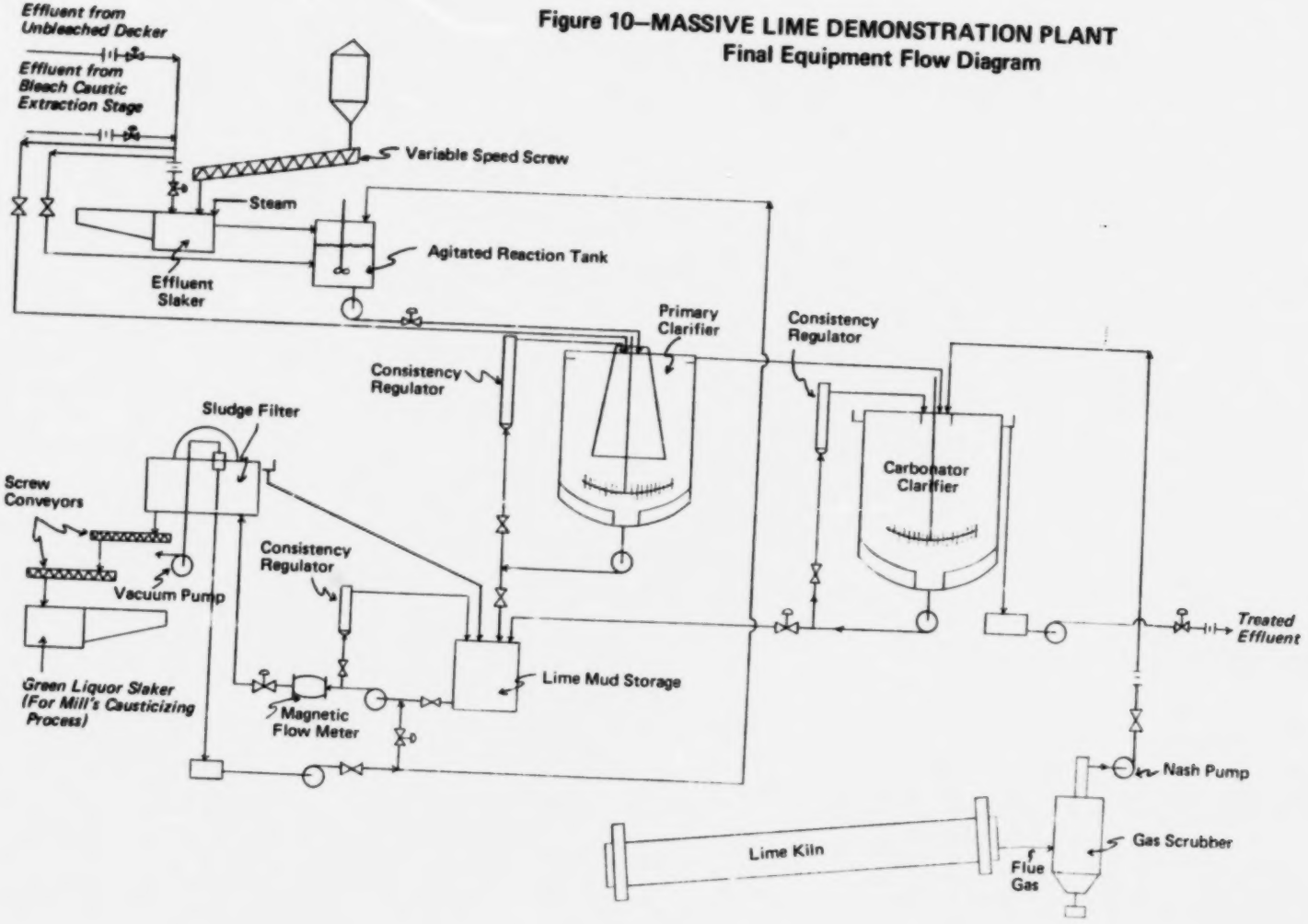
Filtrate from the sludge filter initially was put into the carbonator clarifier. However, after experimenting with addition of the filtrate to the primary clarifier, reaction tank, and slaker, the reaction tank was chosen as the best point in the process at which the filtrate could be recycled. Addition of filtrate to the slaker was undesirable because of the increased steam consumption required to heat the filtrate.

Finally, the sludge filter was changed from precoat operation to continuous belt operation. This change gave a slight increase in the filter's capacity. Water content of the filter cake was essentially the same with either type of operation.

The Final Plant

Figure 10 is a flow diagram showing the massive lime process demonstration plant as finally operated. The most important changes made in the plant were those made in the primary clarifier. These changes, completed in January 1971, allowed the primary clarifier to produce a clarified effluent and enabled the process to be evaluated as planned. Appendix A is a chronological list of changes made to the original plant, and reasons for the changes.

Figure 10—MASSIVE LIME DEMONSTRATION PLANT
Final Equipment Flow Diagram



SECTION V

OPERATION AND PERFORMANCE OF THE EQUIPMENT

After January 1971, when the major operating problems were solved by equipment changes, it was possible to start evaluating both the equipment's performance and the effectiveness of the massive lime process. The following operating schedule was decided upon and followed for the remainder of the project.

1.	100% bleach caustic extraction effluent	4 weeks
2.	Increasing amounts of kraft decker effluent	3 weeks
3.	50% bleach caustic extraction effluent + 50% kraft decker effluent	4 weeks
4.	Decreasing amounts of bleach caustic extraction effluent	3 weeks
5.	100% kraft decker effluent	5 weeks
	a. Reuse of final effluent with carbonation.	
	b. Reuse of final effluent without carbonation.	
6.	Compare precoat with continuous belt-type operation on the sludge filter.	

The above schedule was followed and sufficient reliable data on the operation were obtained to predict the impact of this process on the pulping operation and to develop significant information for design purposes.

This section discusses important operational parameters and the performance of individual items of equipment—overall effectiveness of the massive lime process is discussed in Section VII.

Slaker

A temperature of 210°F was found to be the most desirable operating temperature. At temperatures much above 210°F, boiling and foaming occurred in the slaker. At temperatures much below 210°F, incomplete slaking of the lime occurred with an increase of rejected material from the slaker.

Once liquid in the slaker reached 210°F, heat evolved from slaking was sufficient to maintain the temperature provided the ratio of incoming effluent to reburned lime was maintained at the proper level. A ratio of 6 gallons of liquid to five pounds of lime was required for typical operating conditions. If temperature of either stream changed however, a change in the ratio or other adjustment was of course required to maintain the heat balance. If the reburned lime feed screw became plugged or partially plugged, temperature in the slaker would drop, and the slaker's temperature controller would add steam. This proved to be a good indication of the condition of the lime feed screw conveyor.

The normal temperature of bleach caustic extraction stage effluent entering the system ranged from 135 to 145°F and the decker effluent ranged from 115 to 125°F.

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Reaction Tank

Proper operation of the reaction tank was essential to avoid foaming problems in the primary clarifier. Agitation in the reaction tank had to be sufficient to prevent settling of the suspended solids. To avoid foaming problems, however, it was essential that; (1) all liquor inlets be below the liquid level of the tank; (2) the tank's agitator be run at the minimum speed that would prevent settling, and (3) the agitator be positioned to avoid air entrainment. Liquid had to be maintained at a level which would not create problems with air entrainment (for example, improper liquid level could cause air entrainment by causing splashing of inlet streams, by changing the agitation, and by causing the discharge pump to suck air).

To reduce lime losses from the system, filtrate from the sludge filter (which contained considerable suspended matter) was pumped into the reaction tank. This tank was the best place for re-admission of these suspended solids.

Primary Clarifier

Several factors affected efficiency of the primary clarifier. Among those factors were: (1) air entrainment or foam, (2) entrance into and design of the center well, (3) design of the overflow into the launders, (4) temperature, (5) composition of the suspended or settleable solids in the slurry, (6) flow rate, and (7) rate of withdrawal of the settled solids. Many of these factors are interrelated.

The first three of the above factors are discussed in Section VI and the original primary clarifier design was modified based on a better understanding of those factors. The last four factors—temperature, composition of solids, flow rate, and withdrawal rate—were evaluated with the modified clarifier.

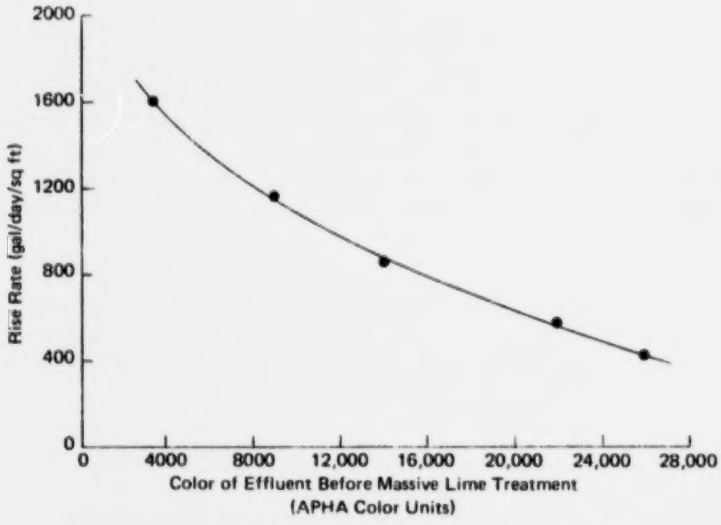
Temperature of the mixture entering the primary clarifier was varied from 150 to 180°F, while treating bleach caustic extraction effluent, and from 136 to 175°F while treating kraft decker effluent. These variations had little if any effect on settling rate of the sludge, but as temperature to the clarifier was raised, more gas bubbles were released. Samples of the flow to the clarifier showed a definite increase in the volume of foam produced at higher temperatures.

The composition of the solids in the primary clarifier slurry was one of the main determinants of settling rate. Differences in solids composition are mainly a function of the color of effluent being treated and the quantity of lime used in the treatment. When an excess of lime was not used, precipitated organic compounds (such as color bodies) formed a voluminous floc which was light and settled very slowly. Even when great excesses of lime were used, settling rate was significantly slower and the settled sludge was less dense as the quantity of organic material increased.

The maximum flow rate (rise rate) at which the primary clarifier could produce a clear effluent was found to be a function of the color of effluent being treated in the system. Figure 11 is a graph of the relationship between rise rate and effluent color.

Figure 11

PRIMARY CLARIFIER MAXIMUM RISE RATE VERSUS
UNTREATED EFFLUENT COLOR



The rate at which settled solids were removed from the clarifier had an effect on both clarity of the unit's overflow and on performance of the sludge filter. Solids content of sludge removed from the clarifier increased as residence time of the settled sludge in the clarifier increased. However, if withdrawal rate was too low for a given flow rate to the clarifier, the sludge bed within the clarifier would deepen until it eventually overflowed the tank. If withdrawal rate was too high, solids content of the sludge would be low and the sludge filter would then be overloaded.

Conditions inside the clarifier were determined by observation of samples taken from four sample lines on the side of the clarifier (Figure 4). Under good operating conditions when the overflow was clear, the following conditions existed; solids content at the bottom sample line was 8 to 10%: solids content at the second sample line was 1 to 3%: the appearance of samples from the third sample line was very cloudy: and samples from the top sample line were hazy. If color of the effluent increased significantly or lime feed became erratic, the following conditions were normally noticed. Solids from the bottom sample line remained about the same; solids in the sample from the second sample port increased to 4 to 8%; the sample from the third line became muddy; the sample from the top line became very cloudy; but the overflow would remain clear to slightly hazy. If the upset condition continued for as long as 4 hours, mud would be present in the overflow. Flow to the system had to be decreased, or withdrawal of settled sludge increased, to correct such upset conditions. Once the sludge bed had risen above the third sample point, it took several hours at a lower flow rate or lower color load to lower the bed to a good operating level.

Carbonator Clarifier

The main factors affecting solubility of calcium hydroxide appeared to be concentration of sodium and chloride ions, temperature and pH. All of these factors also seemed to affect the size and rate of calcium carbonate crystal formation upon carbonation.

The reaction of carbon dioxide (from flue gas) with calcium hydroxide in the solution precipitated calcium carbonate in two physical forms. One of these was a heavy crystalline calcium carbonate which settled rapidly. The other was a very fine precipitate of colloidal nature which would not settle in a beaker in 24 hours. Work done by C. L. Davis and others at Interstate Paper Company, Riceboro, Georgia, indicates that recirculating some of the settled calcium carbonate to the carbonation chamber aids formation of the heavy precipitate and reduces the colloidal calcium carbonate (Reference 10). This recirculation was not tried during this project.

Settled sludge (mostly calcium carbonate) was withdrawn periodically from the carbonator clarifier because the underflow pump and controls were too large to handle the small volume on a continuous basis. This withdrawal took place when sludge concentrations of 20 to 25% were reached.

Table 1 shows calcium removal in the carbonator clarifier was not a function of temperature and pH. Data in the table are grouped to show periods of high, medium, and low concentrations of color, sodium, and chloride in the influent to the massive lime system.

Table 1

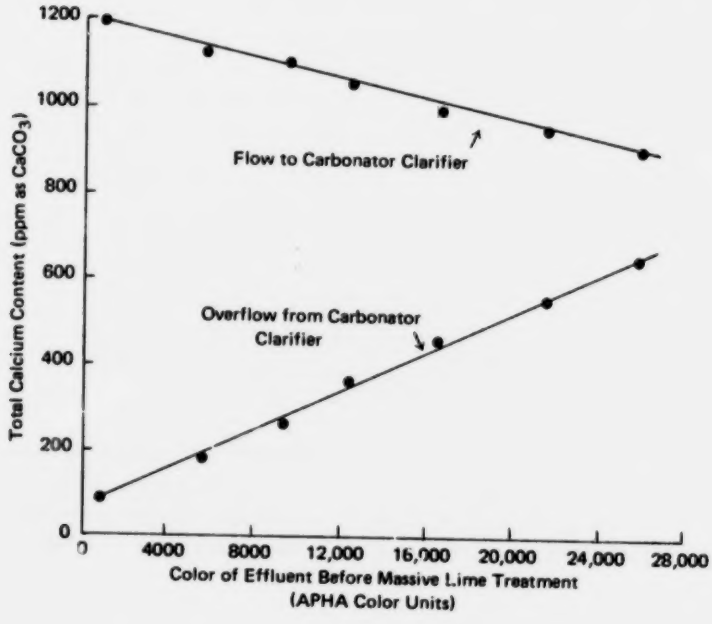
CALCIUM LOSSES FROM CARBONATOR CLARIFIER

Untreated Effluent From Pulp Mill			Overflow From Carbonator Clarifier		Ca (ppm)			
Color, APHA Units	Na, ppm	Cl, ppm	pH	Temp, °F	To Carbonator Clarifier	Removed by Clarifier	From Clarifier Total	From Clarifier Soluble
26,250	1993	1813	12.0	165	663	382	281	61
24,800	1840	1723	11.9	167	390	92	298	52
19,800	1610	1308	12.2	162	336	88	248	100
19,050	1560	1409	11.7	163	362	126	236	40
18,600	1300	1145	12.3	159	400	188	212	44
10,950	1060	846	11.2	159	512	291	221	116
10,500	890	634	11.6	154	468	198	270	21
10,200	983	796	12.2	158	476	263	213	28
8900	910	614	12.2	153	464	336	128	57
8600	820	704	12.0	157	484	238	246	30
1280	545	67	12.3	151	498	465	33	14
1125	480	63	11.2	146	534	444	90	82
730	608	72	11.8	145	477	452	25	20
525	560	79	11.8	140	504	430	74	20
470	520	85	12.5	148	514	476	38	7

Figure 12 shows the concentration of calcium in waters to and from the carbonator clarifier as a function of the original waste water color. Data for these curves were obtained from samples taken under stable conditions when effluent from the primary clarifier was essentially non-turbid and pH in the carbonator was stable. The data indicate that the presence of large amounts of color, sodium ions, and chloride ions affect the calcium content of streams to and from the carbonator clarifier, reducing the efficiency of calcium recovery for higher colored effluent. This is possibly the result of the formation of colloidal calcium carbonate.

Figure 12

CALCIUM CONTENT IN EFFLUENT TO AND FROM THE CARBONATOR CLARIFIER VERSUS UNTREATED EFFLUENT COLOR



Sludge Filter

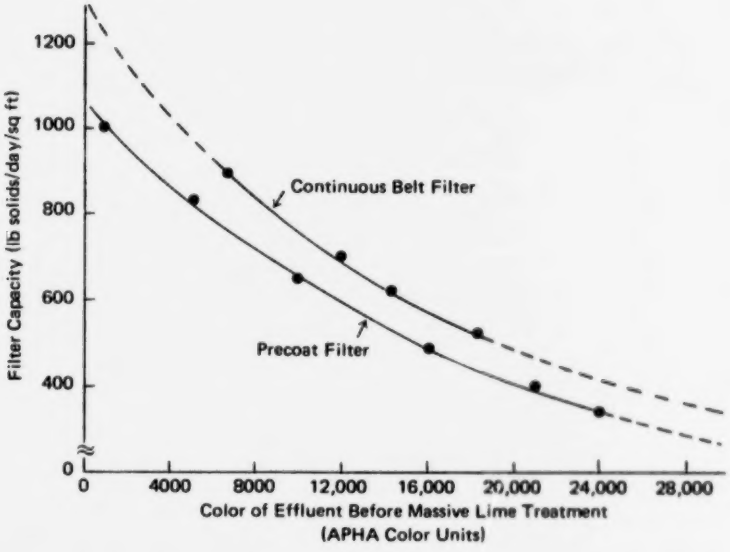
Volume and percent solids of material going to the sludge filter were regulated and flow through the green liquor slaker was controlled based on the quantity of material going to the sludge filter. With this type control, it was essential that overflow from the sludge filter (back to storage tank) be avoided.

The quantity of insoluble organic material in the sludge was probably the most influential parameter governing the dewatering ability of the sludge filter. Even with the weight of inorganic material present being several times more than that of the organic solids, the organics made the sludge slick and slimy and had a tendency to blind over the filter face. As the ratio of organic to inorganic solids was reduced (as lower color effluent was treated), the dewatered cake lost some of its tendency to seal over the filter surface. The cake became more porous, and volume of the discharged cake increased as effluent color decreased. The

sludge filter was initially operated as a precoat filter and was later changed to continuous belt operation for comparison. Capacity of the filter for both types of operation is shown in Figure 13 as a function of the color of effluent being treated by the system.

Figure 13

MASSIVE LIME SLUDGE FILTER PERFORMANCE



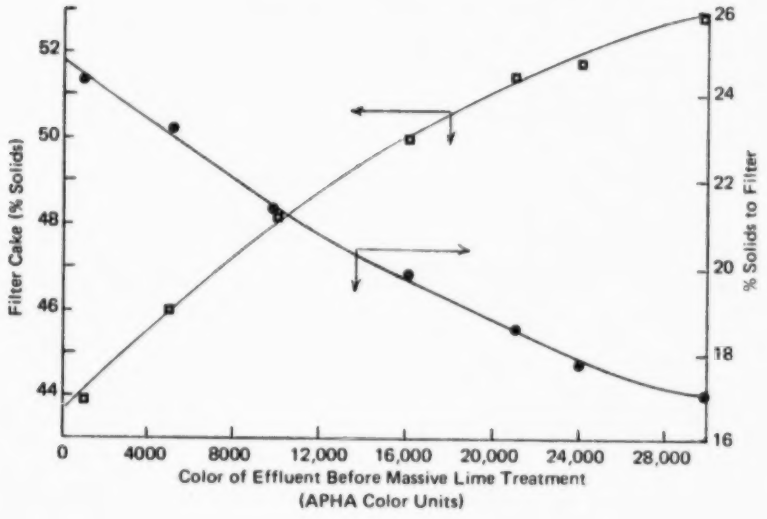
Dewatered filter cake formed from sludge having a high ratio of organics to inorganics was thinner and more dense than cake formed from sludge with lesser amounts of organics. Although filter cake with high organics content was thin, it had very low porosity. Such filter cake was drier (contained less water) than filter cake with lower organic solids content (Figure 14).

A slightly drier cake could be discharged from the filter at slower drum speeds with very little effect on capacity of the filter. Drier solids were beneficial in reducing water load and steam requirements in the pulp mill cooking liquor system.

Deposits built up on the wires which formed the filter face during precoat operation and on the fibers of the belt upon which the cake was formed when the filter was operated as a belt filter. This build-up restricted the flow of liquid through the filter and reduced the filter's capacity. Back washing of the wire or cloth was necessary about every 6 hours of continuous operation. Back washing did not remove all of the deposits, but did remove enough in about 5 revolutions of the washer to allow the filter to perform satisfactorily. Soon after back washing, vacuum in the filter built up to 22 inches of mercury (the maximum). A vacuum break was set at this negative pressure to protect the filter drum, hose connections, and associated piping.

Figure 14

SOLIDS CONTENT OF FILTER CAKE FROM SLUDGE FILTER VERSUS UNTREATED EFFLUENT COLOR



About every two days, scale built up until back washing alone did not provide sufficient cleaning of the mesh or cloth for good operation. When this occurred inhibited muriatic acid was used for cleaning. The acid was slowly poured onto the rotating face of the filter. The entire surface of the wire or cloth was treated. After acid cleaning, the filter was thoroughly back washed to remove solids and residual acid before starting up. The entire acid cleaning procedure required 45 to 60 minutes. (Note: The acid is harmful to human skin and clothing. Fumes from the acid are harmful to the eyes, nose, and throat. Protective clothing, face shields, and absorbent respirators should be used by personnel handling this acid.)

Immediately after back washing and particularly after acid cleaning, solids content of the filtrate sometimes reached 7%. This would decrease to 0.5 to 1% solids just prior to back washing. This filtrate with its suspended and dissolved solids was too valuable to waste. Therefore it was recirculated to the reaction tank.

SECTION VI

EFFECTIVENESS OF THE MASSIVE LIME PROCESS

Before the massive lime plant started operating, analytical tests were made at regular intervals on green liquor, white liquor, lime, lime mud, black liquor, and unbleached pulp. Also, the bleach caustic extraction stage effluent and unbleached decker effluent were analyzed regularly to obtain a history of the variation in concentration of components. These same tests were made during the operating period. A comparison of data from the two periods indicated no discernable change in mill operating conditions, although normal fluctuations occurred during both periods.

Appendix B describes the analytical tests used in this project while detailed operational test data are given in Appendix C.

Sources of Color In Kraft Pulp Mill Effluents

The color and organic carbon content is different for effluents from various processes within a mill. Data for some of the effluents are shown in Table 2. As seen from data in this table, the two most highly colored effluents are from (1) the pulping and recovery area (including the unbleached decker) and (2) the bleach caustic extraction stage. The ratio of color to organic carbon also is different for different effluents.

Table 2

COLOR AND ORGANIC CARBON CONTENT OF MILL EFFLUENTS

Effluent source	APHA Color Units	Total Organic Carbon, ppm	Ratio of Color to TOC
Pulping and recovery area	2566	370	6.9 : 1
Chlorination stage of bleaching	1388	358	3.9 : 1
Bleach caustic extraction stage	21,550	1446	14.9 : 1
Paper mill	346	104	3.3 : 1
Combined mill sewer[1]	2401	316	7.6 : 1
Stabilization basin (combined mill sewer in holding basin for 4 months)[1]	2181	217	10.0 : 1

[1]Contains all of the individual effluents from the mills.

Variations of ± 50% were observed in color in the combined mill discharge. Variables of at least this extent were noticed in each of the individual process discharges.

To convert the color data to a weight basis each color unit was assumed equal to 1 ppm. That is, a 10,000 color unit liquid was assumed equal to 10,000 ppm of color present. Using this procedure, the color discharged from pulping and bleaching operations (as pounds per ton of bleached pulp) was calculated, and the results are shown in Table 3 for periods when either pine or hardwood pulp was being produced. The color loads listed in Table 3 do not include losses from the evaporation process, the recausticizing process, or the cooking

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process. Brown stock washing and bleaching processes are included. Data in Table 3 show that the bleach caustic extraction stage produces the greatest load of color bodies of any of the pulp mill effluents.

Table 3

COLOR LOAD PRODUCED BY PULP MILL SOURCES

Effluent source	Pine Pulp — Color, lb/Ton	Pine Pulp — Percent	Hardwood Pulp — Color, lb/Ton	Hardwood Pulp — Percent
Pulp mill – general	31	4.6	32	6.2
Chlorination stage	130	19.1	93	18.1
Caustic extraction stage	495	72.8	354	68.7
Rest of bleaching process	24	3.5	36	7.0
Total	680	100.0	515	100.0

Paper mill color losses on samples with pH adjusted to 7.6 and allowed to settle seldom exceeded 10 pounds of color per ton when producing bleached paper which contained only small amounts of tinting dyes. However, color far exceeded this amount when making deeply colored papers.

Performance Of The Massive Lime Process

The demonstration plant was used to treat bleach caustic extraction stage effluent, unbleached decker effluent, and various combinations of the two effluents. Tables 4, 5, and 6 summarize the analytical tests made on these effluents before and after treatment. Appendix C gives the daily averages from which the overall ranges and averages shown in Tables 4, 5, and 6 were determined.

Table 4

ANALYTICAL TESTS ON BLEACH CAUSTIC EXTRACTION STAGE EFFLUENT BEFORE AND AFTER MASSIVE LIME TREATMENT

(Data from period: February 8 to March 10, 1971)

	Feed to Massive Lime Process			Primary Clarifier Overflow			Discharge from Massive Lime Process		
	Av	Min	Max	Av	Min	Max	Av	Min	Max
Flow rate, gpm	170	100	250	–	–	–	–		–
APHA color units	21,546	13,000	29,000	1459	1095	1815	1265	565	2000
Suspended solids, ppm	27	10	60	185	20	880	195	10	410
Dissolved solids, ppm	5748	4580	7900	–	–		5409	3860	6910
Total carbon, ppm	1520	1125	1925	–	–		448	290	680
Inorganic carbon, ppm	75	25	125	–	–	–	81	10	250
Total organic carbon, ppm	1446	1050	1850	–	–	–	373	260	530
Ca, ppm, total	43	23	70	490	240	937	222	44	324
soluble	–	–	–	–	–	–	105	28	264
Cl, ppm	1471	1035	2051	–	–		1430	1085	1872
pH	10.0	8.6	11.0	–	–		12.5	11.7	13.2
BOD_5, ppm	353	288	444	–	–	–	264	198	342

Table 5

ANALYTICAL TESTS ON 50/50 MIXTURE OF CAUSTIC EXTRACTION EFFLUENT AND DECKER EFFLUENT BEFORE AND AFTER MASSIVE LIME TREATMENT

(Data from period: April 7 to May 15, 1971)

	Feed to Massive Lime Process			Primary Clarifier Overflow			Discharge from Massive Lime Process		
	Av	Min	Max	Av	Min	Max	Av	Min	Max
Flow rate, gpm	395	300	500	–	–	–	–	–	–
APHA color units	10,043	5350	17,500	522	230	1070	451	185	800
Suspended solids, ppm	44	10	100	76	10	420	196	40	510
Dissolved solids, ppm	3129	2220	3800	–	–	–	3343	2440	4420
Total carbon, ppm	847	550	1225		–		309	140	620
Inorganic carbon, ppm	53	25	100	–	–	–	64	10	150
Total organic carbon, ppm	798	525	1150	–	–	–	245	110	470
Ca, ppm, total	30	18	48	469	108	790	193	85	394
soluble	–	–	–	–	–	–	70	20	169
Cl, ppm	614	463	815	–	–	–	605	518	765
pH	10.2	9.4	11.4	–	–	–	12.2	11.2	13.2
BOD_5, ppm	300	246	390	–	–	–	223	186	276

Table 6

ANALYTICAL TESTS ON UNBLEACHED KRAFT DECKER EFFLUENT BEFORE AND AFTER MASSIVE LIME TREATMENT

(Data from period: May 29 to July 2, 1971)

	Feed to Massive Lime Process			Primary Clarifier Overflow			Discharge from Massive Lime Process		
	Av	Min	Max	Av	Min	Max	Av	Min	Max
Flow rate, gpm	542	525	550	–	–	–	–	–	–
APHA color units	900	455	2360	225	135	295	234	160	335
Suspended solids, ppm	101	20	710	73	10	390	63[1]	10[1]	150[1]
Dissolved solids, ppm	1098	610	1490	–	–	–	1899[1]	1200[1]	2870[1]
Total carbon, ppm	283	110	740	–	–	–	146	40	250
Inorganic carbon, ppm	15	10	30	–	–	–	21	10	70
Total organic carbon, ppm	268	100	720	–	–	–	126	60	240
Ca, ppm, total	22	11	36	509	420	599	169[1]	24[1]	558[1]
soluble	–	–	–	–	–	–	145[1]	7[1]	546[1]
Na, ppm	277	160	525	–	–	–	525	362	675
Cl, ppm	71	35	221	–	–	–	73	35	110
pH	9.7	9.0	10.9	–	–	–	12.0	11.2	12.8
BOD_5, ppm	138	96	192	–	–	–	75	50	117

[1] Data shown include periods with and without carbonation. Average data for periods either with or without carbonation are:

	With Carbonation	Without Carbonation
Suspended solids, ppm	75	22
Dissolved solids, ppm	1638	2488
Ca, ppm, total	78	514
soluble	51	505

Color Removal

Color removal ranged from 90 to 97%, with the overall average being between 94 and 95% removal. Data showing color removal and organic carbon removal achieved when various mixtures of effluents were treated are given in Table 7.

Table 7

COLOR AND ORGANIC CARBON REMOVAL

Composition of Treated Effluent		Effluent Color (APHA Color Units)			Organic Carbon Content (ppm)		
Bleach Caustic Extraction Stage Effluent, %	Kraft Decker Effluent, %	Before Treatment	After Treatment	Color Removal, %	Before Treatment	After Treatment	Organic Carbon Removal, %
100	0	21,546	1265	94.2	1446	373	74.2
67	33	14,325	745	94.8	1016	253	75.1
60	40	12,125	594	95.1	905	248	72.6
50	50	10,043	451	95.5	798	245	69.3
33	67	6612	331	95.0	569	183	67.8
20	80	4660	298	93.6	450	173	61.5
0	100[1]	1640[1]	140[1]	91.5[1]	270[1]	120[1]	55.6[1]
0	100[2]	900[2]	234[2]	74.0[2]	268[2]	126[2]	53.0[2]

[1]Very little paper mill white water reuse in decker pulp washing and make-up water.

[2]Practically all water used in decker system was white water from paper mill.

A little over 3 months before conclusion of the massive lime project, the mill substantially increased reuse of paper machine white water in the decker system. Practically all wash water, dilution water, and make-up water used in this system was paper mill white water. When this was done there was a significant reduction in color removal when treating unbleached decker effluent in the massive lime system (last line of Table 7).

The reduction in color removal efficiency when using a maximum flow of paper mill white water was probably due to papermaking dyes which were not insolubilized by treatment with calcium hydroxide. Precipitation of colored organic compounds by alum present in the paper mill white water may have caused some color bodies to remain with the pulp thus reducing the decker effluent's color.

Organic Carbon Removal

Organic compounds account for most of the BOD as well as most of the color present in pulp mill waste waters. Data on organic carbon content of effluent before and after massive lime treatment are given in Table 7. From 55 to 75% of the incoming organic carbon was removed by the massive lime treatment, with percent removal generally increasing with higher colored effluent.

BOD Reduction

BOD removal varied from 25 to 45%, with the lower efficiency being from waters containing

the most color and organic material (bleach caustic extraction effluent). The absolute amount of BOD removed decreased very little with large decreases of BOD in the untreated samples. Reasons for this type behavior were not determined. BOD determinations were not made on daily samples as were many of the other tests. Therefore, the BOD data are not as complete or as reliable as the other data.

Table 8 shows average BOD determinations on various waters treated in the massive lime plant.

Table 8

EFFECTS OF MASSIVE LIME TREATMENT ON BOD

	BOD_5 (ppm)			
Source of Sample	**Before Treatment**	**After Treatment**	**Removed**	**Removal, %**
100% bleach caustic extraction stage effluent	353	264	89	25.3
50% extraction stage effluent/50% decker effluent	300	223	77	25.7
100% unbleached decker effluent	138	75	63	45.7

Other Considerations

Although the massive lime process reduces color, organic carbon, and BOD in the waste waters treated, the discharge from the massive lime plant is hot and highly alkaline. It will have to be cooled and at least partially neutralized before being discharged from the mill.

Temperature can be reduced by many methods. If there is sufficient storage capacity between the massive lime plant and the receiving stream, natural evaporation will do an acceptable job.

Neutralization can also be accomplished in many ways. Two of these which are available in a pulp and paper mill are:

1. additional carbonation after the massive lime system with flue gases from the lime kilns or power boilers.
2. dilution or combination of this effluent with waste waters having a low pH, such as the bleach plant chlorination stage effluent or the paper mill effluent.

Mineral solids such as sodium chloride, sodium sulfate, and sodium carbonate are greater in the discharge from the system than in the original waste water.

Material Balances Around System

Data obtained from operation of the demonstration plant were used to calculate material balances around the system. Results of material balances for treatment of bleach caustic

extraction effluent, kraft decker effluent, and a 50/50 mixture of these two effluents are recorded in Figures 15, 17, and 16, respectively. The following assumptions were used in calculating the balances:

1. Composition of reburned lime is; 85.0% calcium oxide, 13.3% inerts, 1.7% soluble sodium compounds (as sodium carbonate), and the remainder is inactive material too light or fine to be removed at the slaker.

2. Organic compounds which combine with calcium do so at a ratio of 1 part calcium to 8 parts organic material.

3. All organic compounds which are reacted with calcium leave the system with the sludge.

4. There are no losses from the system except at the slaker, the carbonator clarifier overflow, and in sludge discharged from the filter.

5. The filtrate from the sludge filter with its dissolved and suspended solids is a constant in the material balances.

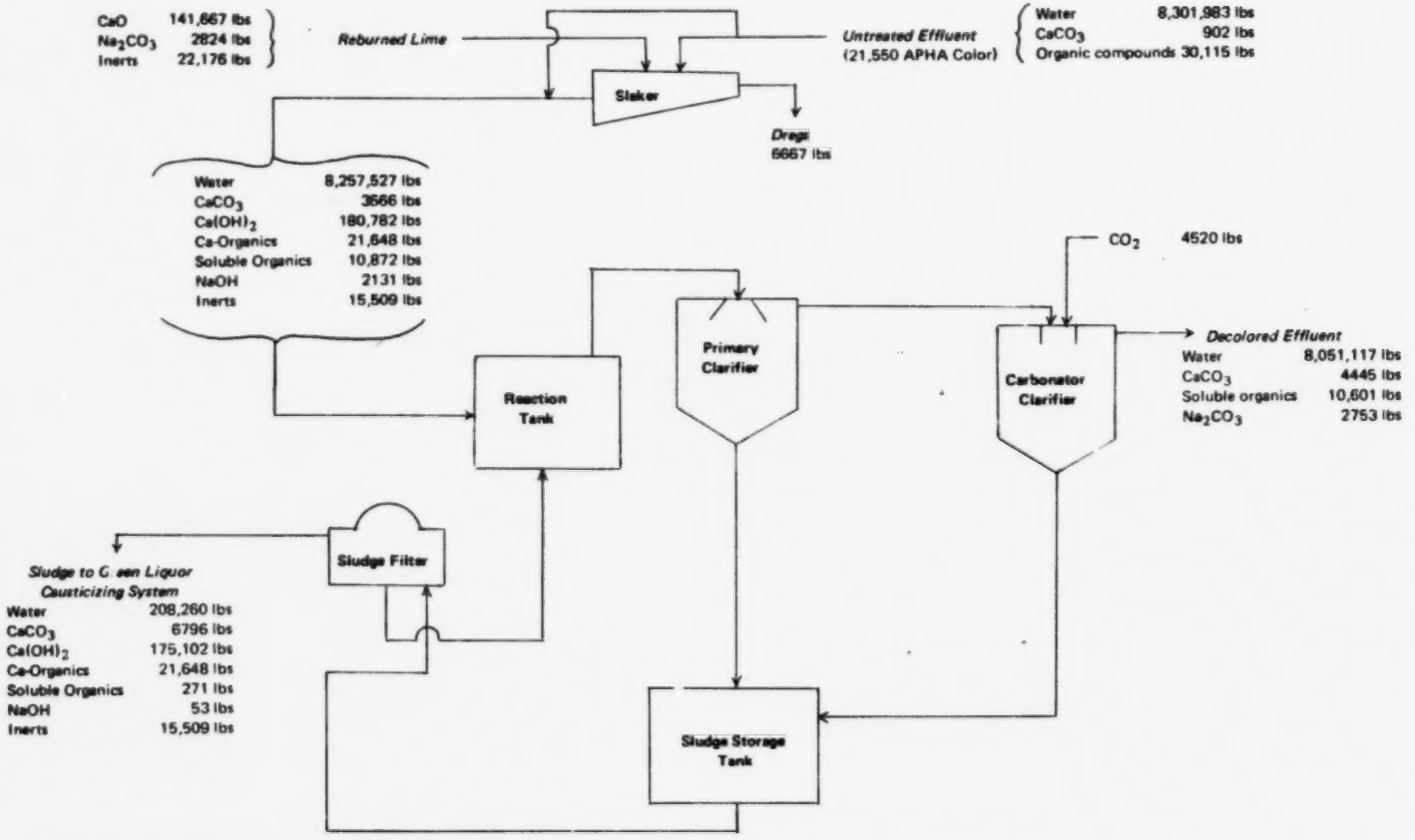
6. The ratio of weight of organic carbon to total organic compounds is that which is found in NCASI Bulletin No. 239 (Reference 9) and is different for the compounds which can be precipitated and those which can not be precipitated.

   Insoluble organic compounds = 0.4647 carbon.

   Soluble organic compounds = 0.3431 carbon.

Figure 15

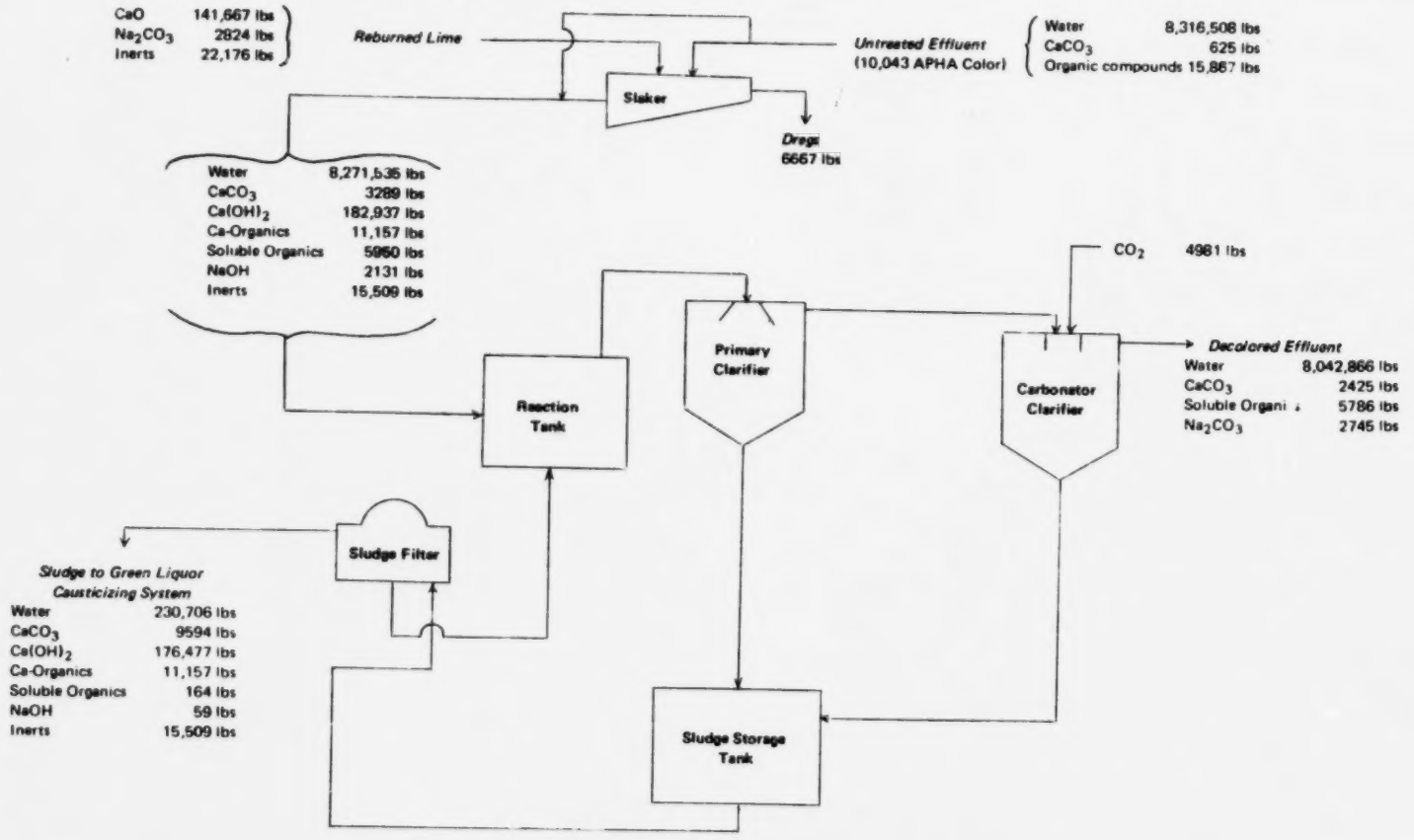
MATERIAL BALANCE FOR MASSIVE LIME PLANT TREATING 100% BLEACH CAUSTIC EXTRACTION STAGE EFFLUENT



Basis: 1,000,000 gallons of effluent to be treated

Figure 16

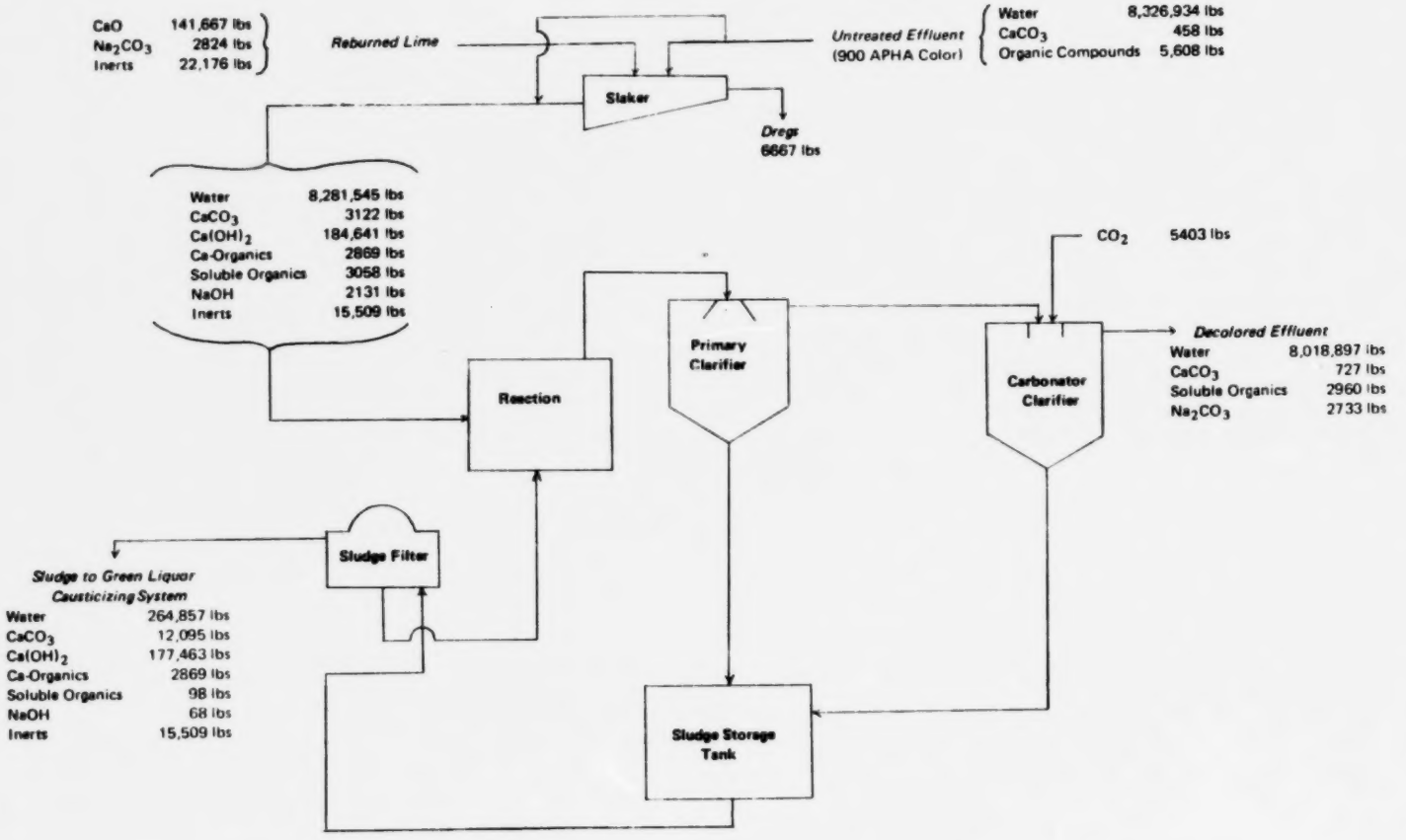
MATERIAL BALANCE FOR MASSIVE LIME PLANT TREATING 50% BLEACH CAUSTIC EXTRACTION STAGE EFFLUENT AND 50% UNBLEACHED DECKER EFFLUENT



Basis: 1,000,000 gallons of effluent to be treated

Figure 17

MATERIAL BALANCE FOR MASSIVE LIME PLANT TREATING 100% KRAFT UNBLEACHED DECKER EFFLUENT



Basis: 1,000,000 gallons of effluent to be treated

SECTION VII

IMPACT OF THE MASSIVE LIME SYSTEM ON NORMAL PULP MILL OPERATIONS

Recovery and reuse of lime is an essential step in the massive lime process. This reuse is accomplished by replacing lime used in the pulp mill's causticizing process (Figure 2) with sludge (mainly calcium hydroxide and water) from the massive lime system. Use of this sludge however, changes equilibrium conditions for the mill's causticizing and black liquor recovery process. Water and organic materials not normally added to the causticizing process are introduced with the massive lime system sludge. In addition, with the normal causticizing system, some water is consumed and heat is liberated when lime is added and reacted with water to produce calcium hydroxide. This does not occur when massive lime system sludge is used for causticizing.

To determine the effects of these changes on the mill's processes for cooking liquor preparation and cooking chemical recovery, the material balances shown in Figures 18 and 19 were calculated. Figure 18 shows results of a material balance for the normal mill system, while Figure 19 shows results of a material balance for the system with massive lime system sludge used for causticizing. Quantities of materials shown in Figures 18 and 19 are the amounts required for each ton of unbleached pulp produced. Since pulp mill cooking chemicals are a complex mixture of various sodium and sulfur compounds (predominantly sodium sulfide, sodium hydroxide, and sodium carbonate), the sodium compounds have been expressed as the equivalent amount of sodium oxide (Na_2O), as is commonly done within the paper industry.

Many assumptions were necessary to make the material balances. The following are the major assumptions on which the calculations were based.

1. Total cooking chemical required per ton of unbleached pulp is equivalent to 836 pounds of sodium oxide. This is equivalent to 675 pounds of active cooking chemical.
2. When not using massive lime system sludge for causticizing, cooking liquor (that is white liquor) concentration is equivalent to 0.1366 gram of total sodium oxide per gram of water.
3. Of the total unclarified white liquor entering the white liquor clarifier, 80% of the liquid overflows as clarified white liquor while 20% of the liquid is discharged from the bottom of the clarifier with the calcium carbonate mud.
4. Normally 550 pounds of reburned lime are used for causticizing green liquor, and reburned lime is normally 85.0% calcium oxide, 13.3% inerts, and 1.7% sodium carbonate.
5. All soluble soda in the reburned lime is in the form of sodium carbonate.
6. Heat required in the normal slaking and causticizing operations is supplied by the exorthermic reaction of lime with water to produce calcium hydroxide.

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Figure 18

MATERIAL BALANCE FOR NORMAL PULP MILL LIQUOR PREPARATION AND SPENT LIQUOR RECOVERY SYSTEM

Basis: 1 Ton of Unbleached Pulp

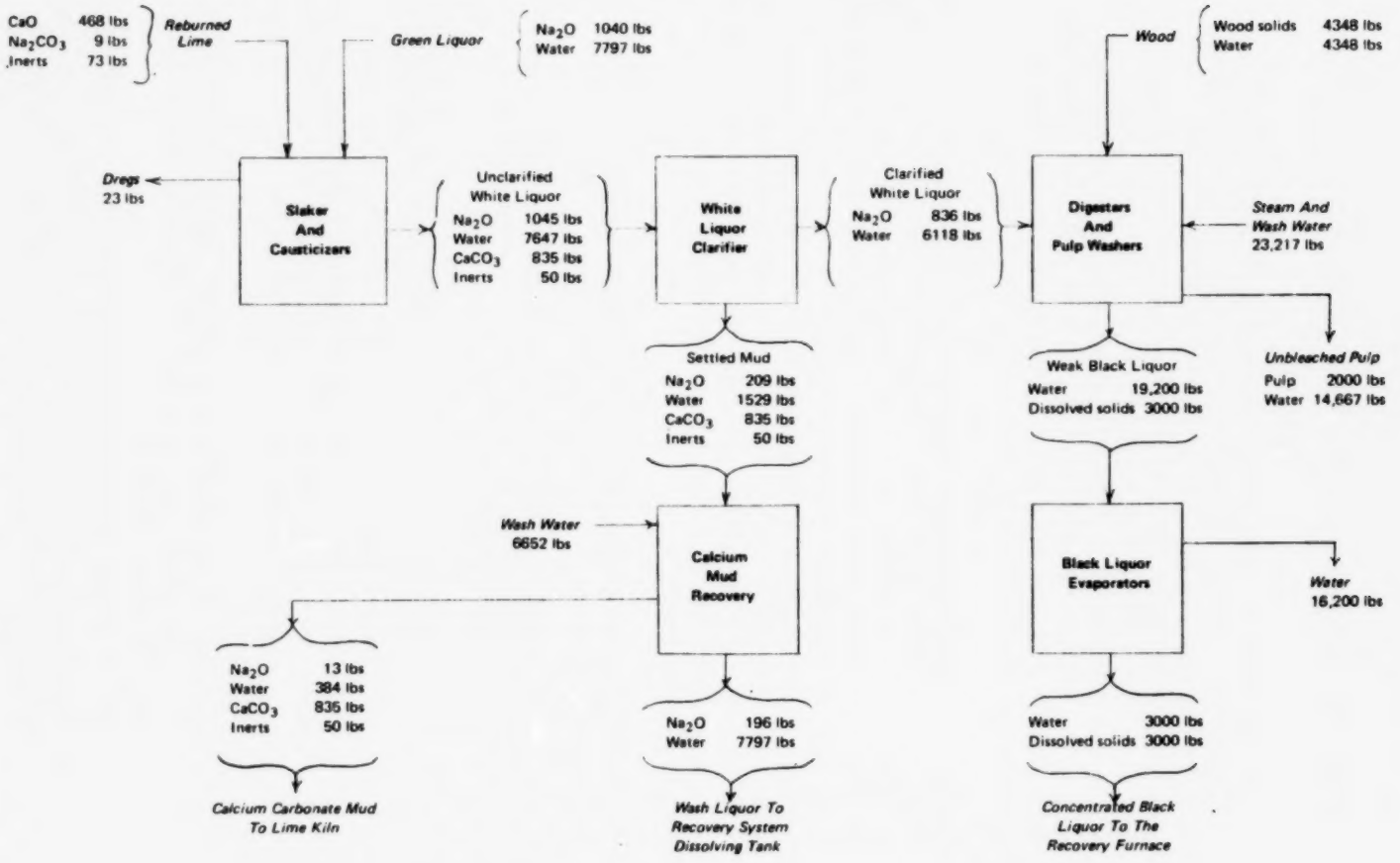
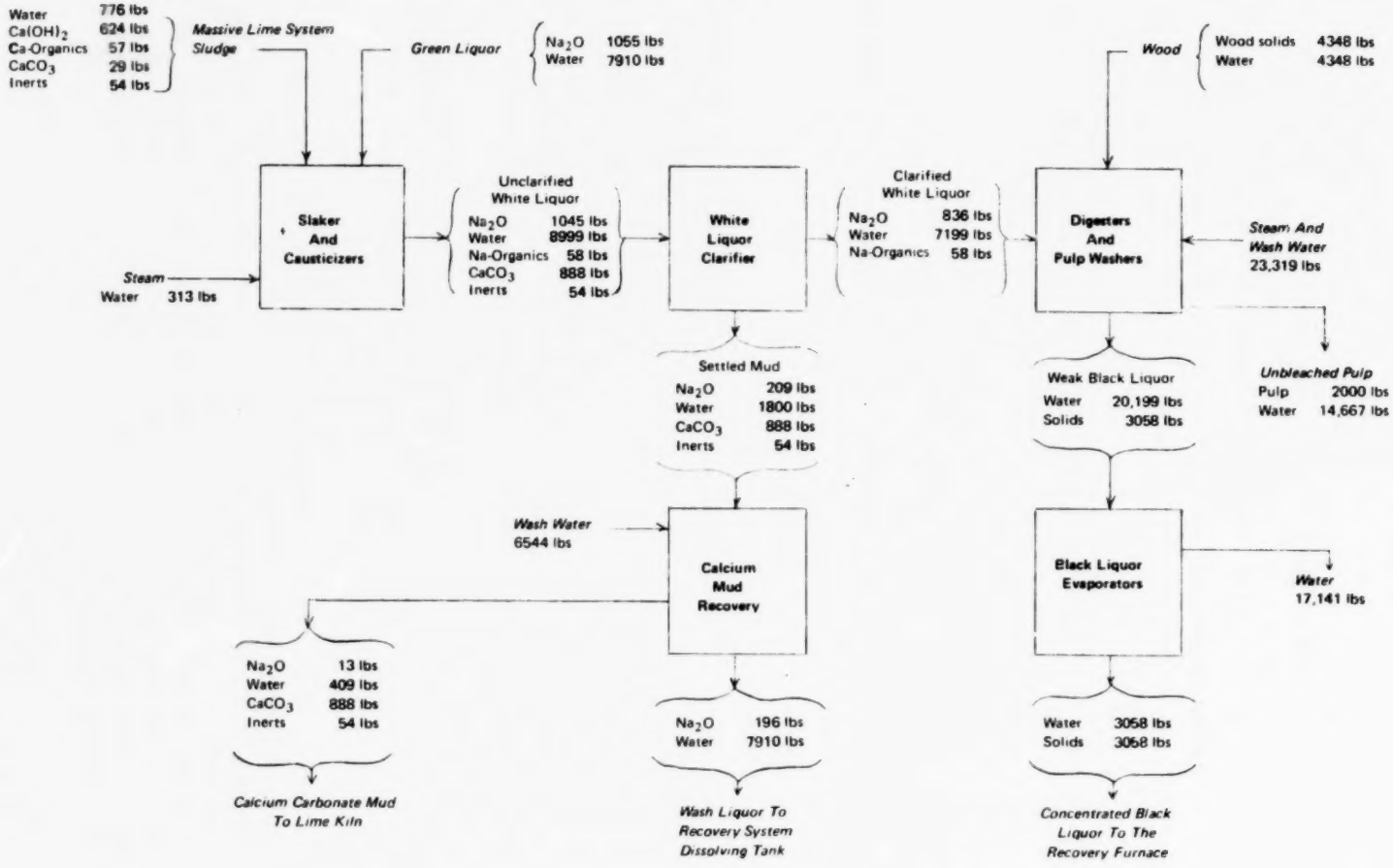


Figure 19

MATERIAL BALANCE FOR PULP MILL LIQUOR PREPARATION AND SPENT LIQUOR RECOVERY SYSTEM USING MASSIVE LIME SYSTEM SLUDGE FOR CAUSTICIZING

Basis: 1 Ton of Unbleached Pulp



NOTE: The composition of massive lime system sludge used in this balance is representative of what would be available at the hypothetical mill described in Section IX.

7. Green liquor concentration is such that there are 7.497 pounds of water present for each pound of total sodium oxide, and the mill normally operates at the maximum green liquor concentration it can handle.
8. Pulp yield is assumed to be 46%, meaning that 46 pounds (dry basis) of pulp are produced for every 100 pounds of wood (dry basis) cooked.
9. Wood entering the system is 50% water and 50% "wood solids."
10. Unbleached pulp is discharged from the system (from the pulp washers) at 12% consistency.
11. The amount of water used to wash the unbleached pulp varies with the total amount of sodium charged to the digester, so that wash water varies 1.25 gallons for a change in sodium equivalent to one pound of sodium oxide.
12. Normally, 3000 pounds of black liquor solids are generated per ton of unbleached pulp, and normal weak black liquor concentration is 13.5% solids.
13. Concentrated black liquor discharged from the evaporators is controlled to 50% solids.

Slaker And Causticizer

When using massive lime sludge for causticizing, the amount of green liquor processed through the pulp mill's causticizing system must be increased approximately 13.6 gallons per ton of pulp, an amount equivalent to an increase of 15.1 pounds of sodium oxide per ton of pulp. This increase is necessitated by two factors. First, the calcium-organic compounds entering the system with the massive lime sludge will react to consume sodium (calcium combined with the organics will be replaced with sodium). Second, sodium compounds previously obtained from the reburned lime are not available from the massive lime system sludge and must be replaced with sodium compounds obtained by increasing green liquor flow.

The slaking reaction (conversion of calcium oxide to calcium hydroxide) in the normal liquor making process consumes 149.7 pounds of water per ton of pulp. This does not occur when massive lime system sludge is used for causticizing. As a result, a more dilute white liquor is produced when using massive lime system sludge.

To equal the causticizing ability of 550 pounds of reburned lime, 1540 pounds of massive lime sludge at 49.6% solids were required. Average temperature of the sludge was 125°F. Therefore, to heat the sludge and green liquor to causticizing temperature 313 pounds of steam were used. Condensation of this steam in heating the liquor also reduced white liquor concentration. The amount of steam required was calculated by assuming green liquor concentration was approximately 18% solids, the heat capacity of green liquor was approximately 0.93 BTU/lb -°F, and the heat capacity of massive lime sludge solids was approximately 0.2 BTU/lb -°F:

- $\frac{\text{(7910 lb water in green liquor)}}{\text{(0.82 lb water/pound green liquor)}} \times \frac{\text{(210 - 185°F change in temp)}}{\text{(970 BTU/lb steam)}} \times \text{(0.93 BTU/lb -°F)} = 231.2$ lb of steam to heat the green liquor
- $\frac{\text{(776 lb water in sludge)}}{\text{(970 BTU/lb steam)}} \times \text{(210 - 125°F change in temp)} \times \text{(1.0 BTU/lb -°F)} = 68.0$ lb of steam to heat water in the sludge
- $\frac{\text{(764 lb solids in sludge)}}{\text{(970 BTU/lb steam)}} \times \text{(210 - 125°F change in temp)} \times \text{(0.2 BTU/lb -°F)} = 13.4$ lb of steam to heat sludge solids

Total steam required = 313 lb

Net effect of all the above factors is to increase water input to the causticizing system by the following amounts:

113 lb due to increased green liquor flow
776 lb entering with sludge
150 lb not consumed in slaking reaction
313 lb from condensation of steam used for heating.
1352 lb = total increase (or 162 gallons) per ton of pulp

This increased water input caused white liquor produced by the system to contain a significantly lower concentration of cooking chemicals.

Foaming problems were created in the slakers, causticizers, clarifiers, and mud washers when massive lime sludge was used for causticizing. Foam overflowed these vessels and tanks causing excessive losses of soda and lime until corrective action was taken. Addition of defoamer to liquor discharged from the slaker controlled the foam (0.5 pound of defoamer per ton of pulp was required).

White Liquor Clarifier

The overall increase in volume of liquid conveyed to the white liquor clarifier was 162 gallons per ton or 17.7%. This means that to do a comparable job of white liquor clarification, the white liquor clarifier's size (surface area) must be increased 17.5 to 20% when using massive lime system sludge for causticizing.

For the material balances shown in Figures 18 and 19, it was assumed that all settleable solids from the white liquor clarifier go to the mud washer, and all organics, from the massive lime sludge go with the white liquor to the digesters. Since the cooking liquor chemicals and wash liquor eventually are recycled to the green liquor, this assumption represents the net effect after equilibrium conditions are established.

White liquor made by causticizing green liquor with filter cake from the massive lime process is black in color, approximately 15% weaker in concentration, and has a higher chloride content than normal white liquor.

The black coloration of the liquor has little or no effect on the cooking of wood chips. Black coloration is caused by basically the same organic compounds that are found in normal weak black liquor used to make up the necessary liquor volume in batch digesters. The black color

however, does interfere with the normal analytical tests used for determination of white liquor composition, since visual observation of the color change of pH indicators is normally used to determine end points for the titration. Instead of titrating to visual color changes electrical pH meters should be used for this analysis.

Mud Washing

The volume of mud handled in the mud washing system increased about 16% and solids to the mud washing system increased by 57 pounds per ton of pulp or 6.4%. Therefore the lime mud filter capacity would have to be increased 6.4%.

To keep the system in balance, water for mud washing would be decreased 16 gallons per ton of pulp. This decrease should result in greater soda losses from the mill when using the massive lime sludge; however, loss of this soda was not considered in the balances shown in Figures 18 and 19.

Lime Kiln

Solids handled in the lime kiln will be increased by 57 pounds per ton of pulp or 6.4%. This requires an increase in kiln capacity as well as an increase in fuel consumption.

A rotary lime kiln with 70% solids feed will require approximately 9,000,000 BTU per ton of reburned lime. Therefore, fuel consumption will increase an amount equal to 576,000 BTU per ton of reburned lime when the massive lime system is used. This results in an increase in fuel consumption equal to:

$$\bullet \quad \frac{(576{,}000 \text{ BTU})}{(2000 \text{ lb of reburned lime})} \times \frac{(550 \text{ lb reburned lime})}{(1 \text{ ton of pulp})} = 158{,}400 \text{ BTU per ton of pulp}$$

Digesters And Washers

Because of significantly lower concentration of cooking chemicals (approximately 15% lower) in the white liquor, the volume of liquor required to furnish the cooking chemicals to the digester is greatly increased. In the case of batch digesters, this means less weak black liquor would be used for volume make up. In the case of continuous digesters, the actual cooking chemical concentration in the digester is reduced. Therefore larger liquor circulating pumps and possibly somewhat larger cooking vessels may be required for continuous digester systems.

Flow to the pulp washers will increase by 134 gallons per ton of pulp. This may not require larger washers but will require an increase of 5.8% in their hydraulic capacity (liquor removal ability) and pumping capacity. Discharge of black liquor solids from the washers will increase from the normal 3000 pounds to 3058 pounds of solids per ton of pulp. Also, the concentration of solids in the weak black liquor will decrease from 13.5 to 13.1%.

Evaporators

The flow of weak black liquor to the evaporators will increase from 22,200 pounds to 23,257 pounds per ton of pulp. Discharge of strong black liquor from the evaporators will remain at 50% solids, but the discharge of strong black liquor will increase from 6000 to 6116 pounds per ton, while solids discharged in the strong black liquor will be increased to 3058 pounds per ton.

The amount of evaporation required will increase from 16,200 pounds to 17,141 pounds per ton of pulp. With an evaporator efficiency of 4 pounds of evaporation per pound of steam, this increases steam requirements by 235 pounds per ton of pulp. This amounts to an increase in evaporator capacity of 5.8%.

Recovery Furnace

Concentrated black liquor is burned in the mill's recovery furnaces where inorganic chemicals are recovered and heat produced by the burning is used to generate steam. Recovery furnaces are generally sized to process 3000 pounds of black liquor solids per ton of pulp and to produce 3.5 pounds of 850 psi steam per pound of black liquor solids.

Since organic solids removed from bleach plant waste waters are similar (derived from lignins) to normal black liquor solids these solids were assumed to have the same handling characteristics and heating value as normal black liquor solids. If so, the recovery furnace size must be increased 1.9% to handle the increased flow of concentrated black liquor.

This increase in solids can also be expected to increase steam production by 203 pounds of steam per ton of pulp. This steam will essentially offset the additional steam required for evaporation, but will also generate 10.4 kw of electricity in a turbine when reducing the pressure from 850 psi to 60 psi gauge.

Effect On Chloride Concentration

Small amounts of chloride ion enter the mill's liquor system from the wood, sodium sulfate, and lime normally added to the process. Therefore green liquor normally contains 800 or more ppm chloride ion. Massive lime treatment of bleach caustic extraction stage effluent containing 1430 ppm chloride will cause an addition of the equivalent of approximately 20 ppm chloride to the green liquor stream each cycle. This addition would have a tendency to increase chloride concentration to an equilibrium level where chloride losses would equal the amount of chloride added. The amount of chloride introduced with the massive lime sludge is probably small enough to have little effect on corrosion or other detrimental conditions in the liquor system.

Effect On Pulp And Paper

Analyses of pulp and paper showed very little if any difference in the final quality of these products. No appreciable difference was observed in bleachability or bleaching of the pulp. Also, no differences were evident in the pulp's performance on the paper machine and there

were no differences in customer's reactions to the finished product. Using massive lime process sludge as a replacement for reburned lime apparently does not affect finished product quality, however it is possible that long term effects on pulp mill processes or on pulp quality, which did not show up in the demonstration plant project, might be observed in a full-scale installation.

Chemical Losses From The Pulp Mill

A fairly good chemical recovery system in a kraft pulp mill requires a chemical make-up of approximately 125 pounds of sodium sulfate per ton of pulp produced. An equally well closed system on the lime cycle requires 20 pounds of new lime makeup per ton of pulp. Considering that these losses are proportional to the quantity of materials carried throughout the cycle, additional losses due to use of the massive lime process sludge are calculated below.

$$\frac{(1055 - 1040 \text{ lb change in } Na_2O \text{ concentration in green liquor})}{(1040 \text{ lb of } Na_2O \text{ in normal green liquor})} \times (125 \text{ lb } Na_2SO_4 \text{ makeup/ton of pulp}) = 1.8 \text{ lb of } Na_2SO_4 \text{ additional losses per ton of pulp}$$

$$\frac{(955 - 898 \text{ lb change in solids to lime kiln})}{(898 \text{ lb solids to lime kiln normally})} \times (20 \text{ lb lime makeup/ton of pulp}) = 1.3 \text{ lb of lime additional losses per ton of pulp}$$

Reuse Of Treated Effluent

Reuse of treated effluent as the scrubbing liquid in the lime kiln flue gas scrubber (see Figure 2) was evaluated using decolored unbleached kraft decker effluent from the demonstration plant. At a flow rate of 550 gpm to the massive lime system, there was insufficient flow to meet the make-up water requirements for the Springhill, Louisiana mill. Clarified effluent, either carbonated or uncarbonated, was pumped from the carbonator clarifier overflow to the sump feeding the lime kiln's venturi scrubbers. However a 1,000,000 gallons-per-day massive lime treating plant would supply the lime requirements for causticizing enough cooking liquor to produce 281 tons of pulp per day and the same plant would discharge enough treated water to supply a caustic room-lime kiln complex for a 1220 tons-per-day pulp mill.

Soluble organic compounds affected surface tension of the scrubbing liquid sufficiently to cause severe foaming in the lime kiln dust collection system. If foaming was not controlled, the overflow and spillage caused excessive losses of lime and soda. Spray showers were unable to control the foam, so defoamer at the rate of 20 ppm was used. The defoamer added to this liquid and the defoamer added to the white liquor system controlled foam throughout the liquor making and consuming operations. Except for the foaming problem, treated effluent performed as well as water from any other source for flue gas scrubbing and caustic room make-up.

With a massive lime system large enough to provide all the lime required for green liquor causticizing, more water would be treated than could be used for make-up in the caustic room-lime kiln complex. Only 22.5% of the treated water could be utilized, so the rest of the treated water would be available for some other reuse or discharge to the mill sewer. Reuse of this water in the brown stock washing system was not tried. If the total hardness

of the treated effluent can be controlled at less than 100 ppm as calcium carbonate, the water could be used on the brown pulp washers. Addition of defoamer and pitch dispersants would probably be required to make treated decker effluent usable in the washing system.

Reuse of treated effluent would result in the following savings:

1. Retain most of the sodium compounds which are normally lost in the massive lime system.
2. Somewhat reduce the amount of heat required for heating brown stock washer wash water.
3. Save the cost of procuring, treating, and delivering the same quantity of fresh water.
4. Save the cost of disposing of a like quantity of waste water.

Reuse of treated bleach caustic extraction stage effluent would probably present more problems than reuse of treated unbleached decker effluent because of the chlorides present in the bleach plant effluent.

SECTION VIII

CONSIDERATIONS FOR DESIGN OF A COLOR REMOVAL PLANT

The massive lime treatment system should be located adjacent to the mill's normal liquor making operations. This will minimize conveying of lime to the slaker and conveying of massive lime sludge from the filter to the causticizing process. New and reburned lime storage facilities can be utilized by both processes. In case of an interruption in operation of the massive lime system, lime could be slaked and used in the normal manner for causticizing.

Calcium scale builds up quite rapidly in pumps and pipe lines after the slaker. This scale must be removed at least every six months or capacity of the lines will be limited. If possible, the massive lime system flows should be by gravity from the slaker through the carbonator clarifier. This would eliminate pumping, and conveying from one unit to the next could be done through open troughs. The troughs can be easily cleaned without shutting down the system. If gravity feed is not feasible, piping should be as short as possible with the fewest possible bends or elbows.

The entire area should be drained to a sump for collection of spills, washup water, and sample discharges. All of these waste liquids usually contain exceptionally high concentrations of calcium compounds, so chemical losses would be excessive if these liquids are not returned to the system. These recovered wastes should be returned to the reaction tank.

The entire massive lime system should be designed to minimize air entrainment at all points, and to release any gas admitted or generated by the process as soon as possible. This will reduce foaming which is disastrous to the efficiency of the operation.

Effluent Supply Tank For Massive Lime System

A sump, chest, or tank should be provided to collect colored effluent and serve as a supply source for the massive lime system. Baffling between the inlet and outlet of the supply tank can be used to eliminate entrained air. The pump or other means of transporting effluent from the supply tank to the massive lime treatment should have the capacity and flexibility to handle most operational surges without allowing air to get into the effluent.

An accurate flow meter should be installed to indicate and record the flow of effluent to the color removal system.

Slaker And Accessories

Figure 20 shows the relationship between the volume of effluent to be treated and the quantity of lime which must be slaked for a 20,000 ppm effluent treatment. For a 1000 tons-per-day bleached pulp mill, enough lime would normally be available to treat approximately 4.0 million gallons of effluent. If a greater volume of effluent is to be treated, the mill would need increased lime recovery capacity.

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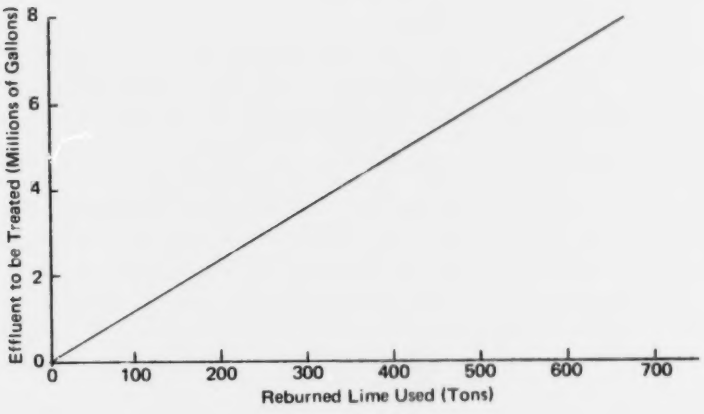
Figure 20

VOLUME OF EFFLUENT TREATED VERSUS LIME REQUIREMENTS FOR MASSIVE LIME PROCESS



Since the quantity of lime to be slaked is directly proportional to the volume of effluent to be treated, this volume must be determined before the slaker can be properly sized. The *maximum* quantity of effluent to be treated should be the governing factor in sizing.

A variable speed feed mechanism is needed to meter lime to the slaker. If the volume of waste water to be treated varies more than ± 10% from the design figure, this meter should be controlled based on the flow of effluent to the system.

To conserve heat, only the minimum quantity of liquid required to slake the lime should be used in the slaker where a temperature of about 210°F is maintained. Once the system around the slaker has come to equilibrium, liquid flow to the slaker can be proportioned and controlled relative to the total liquid flow t· the massive lime system.

Final slaker temperature control by direct steam is recommended. Steam is required during slaker start-up after shut downs exceeding 20 to 30 minutes. A small amount of steam may also be required as trim control due to variations in effluent temperature or reburned lime temperature. A steam flow recorder is a good indicator of pluggage problems in the lime feed mechanism.

The slaker should be equipped with a grit removal device which will discharge into a collection container for easy removal.

Liquid level in the slaker can be controlled by gravity overflow through an inclined pipe or trough.

Reaction Tank

The reaction tank should be designed for 10 minutes retention time based on total flow of

waste water to the system plus 15% of that flow. This additional 15% approximates (1) the flow of filtrate from the sludge filter, (2) seal water from the sludge filter vacuum pump, and (3) the return of spills, wash-up, etc from the area pick-up sump.

All liquid inlets into the reaction tank should be at or below the liquid surface to minimize air entrainment caused by splashing. If gravity flow to the primary clarifier can be used, then an overflow weir can be used to maintain a level in the tank which will avoid splashing of incoming liquid. If however, a pump is required to transport the reaction tank mixture to the primary clarifier, discharge from the pump should be controlled based on the reaction tank level.

An agitator is required to thoroughly mix the various liquids and sludges for uniform reaction between organic compounds and the hydrated lime. Agitator should have design characteristics which will keep suspended solids in suspension without creating a vortex and without generating foam.

Primary Clarifier

The following items should be considered in design of the primary clarifier.

1. A center well sufficiently large to allow for escape of gases and control of the resulting foam with spray showers or surface skimmers.

   The clarifier's center well should be designed for a rise rate of 0.96 feet per minute. This allows for a 33% increase in rate due to turbulence before equalling the observed rise rate (1.28 feet per minute) of air bubbles in treated effluent.

   The center well should extend at least two feet above the water level in the clarifier. Showers should be positioned one foot above the top of the center well so that their spray will cover the surface of the well. One and one-half gallons of shower water per square foot of area should destroy or control normal foaming conditions.

   The center well can be either conical or cylindrical and should extend to at least 50 to 60% of the liquid depth. Liquid entrance into the center well should be below the surface and pointed downward. A deflection plate should be located below the liquid entrance to reduce entrance velocity.

2. A clarifying area sized according to the information given in Figure 11.

   The volume of colored waste water that the primary clarifier can handle is almost entirely dependent on the original color of the liquid. As color increases, the rate of settling (clarification) decreases. Figure 11 shows the relationship between color and the maximum clarifier flow rate in gallons per day per square foot of effective clarification area (total surface area of clarifier—area of center well at overflow level).

   Average color and flow of the effluent to be treated must be known. The

design flow rate should be 25% higher than the expected flow to allow for normal fluctuations associated with operational demands of increased production, increased flows, and occasional spillage.

3. A settled sludge removal system.

   The bottom rake or sludge removal rake, should be designed to deliver to the clarifier's outlet well a volume equal to 20% of the flow to the process. Since the settled solids are very fluid and are easily roused from a settled condition by even medium velocity changes, peripheral speed of the removal rake should not exceed 15 feet per minute. A reputable supplier of such equipment can supply a good design for the removal rake if given the conditions under which the rake will operate.

   Sludge discharged from the clarifier needs to be regulated by both volume and percent settled solids.

4. A clarified liquid overflow device which will not produce channeling in the clarifier.

   Discharge through orifices into the launders worked well in the modified demonstration plant clarifier. A similar arrangement should be satisfactory for any unit.

Carbonator Clarifier

The carbonator clarifier must provide good contact of flue gases containing carbon dioxide with liquid from the primary clarifier; and it should provide a vessel for settling and withdrawing the precipitated calcium carbonate.

The carbonator could be an external vessel or an integral part of the clarifier. In this discussion, it is considered as an integral part of the clarifier.

The bottom of the center well (carbonation area) should extend to a depth not less than 40% nor more than 50% of the liquid depth in the clarifier and the top should extend 1.5 to 2 feet above the liquid level. It is not necessary that the center well be covered. Foam generated by carbonation is the large bubble type and is easily controlled.

Due to turbulence in the carbonator, the point of liquid entrance into the center well is not critical.

The flue gas header and diffuser arrangement shown in Figure 8 provides a practical design. The diffusers or spargers should be equipped with disconnects from the header for easy removal for replacement or cleaning. A flue gas flow of approximately 0.75 to 1.0 cubic foot for each gallon of effluent per minute is required.

Based on the work done in this project, the clarifier should be designed for a liquid rise rate

of 1.5 gallons of water per square foot per minute. However, work done by Mr. C. L. Davis of Interstate Paper Company, Riceboro, Georgia, indicates calcium carbonate crystal growth can be increased by recirculating part of the sludge to the liquid entering the carbonator. Such seeding is reported to give more than 90% recovery of the calcium in the water (Reference 10). As stated in his report, rise rates in the clarifier could be increased by using this procedure.

The sludge removal rake in the carbonator clarifier does not handle nearly as much sludge as the one in the primary clarifier. Also, the settled sludge is denser and the rake speed not so critical. Again, a reputable supplier of such equipment could design a suitable rake when given the operating conditions.

Sludge removed from the carbonator clarifier can either be pumped to the sludge storage tank and then to the massive lime sludge filter, or to the causticizing system mud storage tank which feeds the lime kiln thickener. The calcium carbonate mud improves drainage properties of the massive lime sludge on the sludge filter if the two muds are mixed in the sludge storage tank. But, the mixture puts a slightly heavier load of inactive solids on the slakers, white liquor clarifier, and mud washing equipment in the causticizing system. If completely carbonated, the carbonator clarifier sludge can be sent to the lime kiln and it will not interfere with operation of the lime kiln.

Sludge Storage Tank

This tank is to provide surge capacity between the clarifiers and the sludge filter, and to provide storage space for mud when emergency repairs or breakdowns occur at the massive lime sludge filter or in the caustic room area. This tank should accommodate 3 hours of sludge from the massive lime system. For each 1,000,000 gallons of effluent per day to be treated in the massive lime system, this tank should hold at least 3342 cubic feet of sludge.

An agitator large enough to keep sludge in the tank homogeneous should be provided so a uniform consistency sludge can be supplied to the sludge filter. The tank should be provided with a consistency regulator to control solids content of sludge pumped to the sludge filter.

This storage tank should also be used to collect slurry dumped from the sludge filter vat when back washing or acid cleaning the filter. It will also be used as a collection tank for slurry that overflows the filter vat when the filter becomes overloaded.

Sludge Filter And Accessories

Surface area required for the rotary vacuum filter used in dewatering sludge reclaimed from the massive lime process is a function of the color and organics content of the water being treated. The higher the color of the effluent to be treated, the more difficult and slower the sludge dewaters. Figure 21 shows the relationship between effluent color and the area of filter surface required to dewater sludge per 1,000,000 gallons of effluent treated per day. Since the filter will not operate continuously because of back washing, acid cleaning, emergencies, etc, the area obtained from this graph should be divided by 0.9 for design purposes. In addition to the running time factor, sufficient surface area should be provided for those

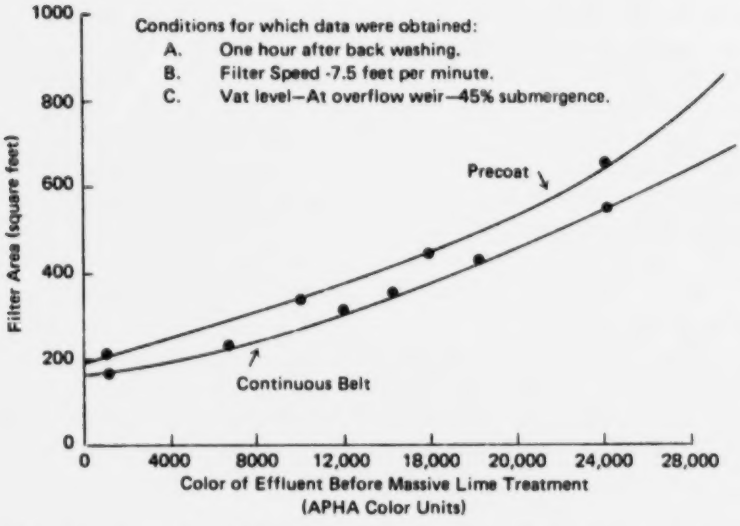
periods of operation when flows or color loads exceed the design capacity due to increased production, etc. The mud storage tank provides space for short time surges but will not overcome increases over a long time period. An additional 25% of filter area should be provided. Therefore total surface area of the sludge filter should be:

(Filter area from Figure 21 ÷ 0.9) x 1.25

Figure 21

SLUDGE FILTER AREA VERSUS EFFLUENT COLOR

Basis = 1,000,000 gallons of effluent treated per day



Back washing the filter should be automatically controlled using properly time sequenced actions for greater efficiency. For best back washing results, shower header sprays should be no more than 2 inches from the washer face. Sprays from the showers should overlap and pressure on the shower header should be about 250 psi. High pressure is more essential than high volume for good back washing.

Acid cleaning should be done mechanically, both for personnel safety and greater cleaning efficiency. The acid cleaning system should include a storage tank for inhibited muriatic acid, acid pump, piping, and a small volume overlapping spray header the width of the filter. The size of the acid storage tank would be governed by capital cost of the storage and the price of muriatic acid.

The filtercake discharged from the sludge filter can be delivered to the green liquor reaction tank or slaker by screw conveyor. Capacity and design of this screw depends on the volume of sludge cake to be moved. When these conditions are known, screw conveyors can be properly sized.

Except for the limited experimentation with reuse of the kraft decker effluent, the final dis-

position of treated waste water from the massive lime process has not been considered. The effluent, before discharge into a receiving stream, would have to be further treated for pH adjustment, temperature reduction, and BOD_5 reduction. Methods of treatment and costs to meet these requirements have not been explored in this project.

Coordination

Probably the most difficult design parameter in incorporating a massive lime system into a normal pulp mill liquor system is that of predicting variations in the demand for causticizing chemical due to production variations. Not including mechanical breakdowns or clothing changes, production rate of an average paper machine can vary ± 25% due to the grade mix assigned to the machine. This variation directly affects the usage of pulp, and in turn, the demands on the liquor and recovery system of the pulp making process.

When production increases, total color (organic compounds) generated in the pulp mill and bleach plant increases. However, the consumption of water does not increase proportinately, thus the color of the effluents tends to increase. Design of a massive lime plant should consider these variations. Sufficient storage for green liquor, white liquor, lime mud, re-burned lime, and massive lime sludge should be provided.

The massive lime system cannot be operated as a water treating plant clarifying a given flow and a given organic load. It must be able to fluctuate to some extent with variation in the mill's production.

SECTION IX

BENEFITS AND OPERATING COSTS FOR A TYPICAL MILL

To illustrate what can be expected from using the massive lime process in a full-scale mill installation, data presented earlier in this report were used to calculate effects of a massive lime process on a hypothetical 1000 tons-per-day mill.

Conditions Assumed For Hypothetical Mill

The mill is assumed to be a new kraft pulp and paper mill producing a 75% pine/25% hardwood bleached pulp. Bleached pulp production is 1000 tons per day so unbleached pulp production is 1136 tons per day. The equivalent of 550 pounds of 85% causticizing power lime is used per ton of unbleached pulp for production of cooking liquor.

Estimated use of fresh water is 20,000 gpm or approximately 29 million gallons per day. The evaporator, brown stock washer, recovery boiler, and black liquor storage systems have catch basins for collection and return of leaks and spills. These systems are of sufficient size to operate efficiently at maximum production with minimum losses.

Total effluent from the mill is approximately 29 million gallons per day. Without massive lime treatment color of the total combined mill effluent is 2631 APHA units. Color loads contributed by various individual streams within the mill are shown in Table 9.

Table 9

CONTRIBUTION OF EFFLUENT SOURCES TO TOTAL MILL EFFLUENT COLOR WITH NO MASSIVE LIME TREATMENT

Source of Effluent	Color Load (lb color/bl ton)	Contribution to Combined Effluent Color (APHA Color Units)
Pulp mill—general	31	129
Paper mill	5	21
Bleach chlorination stage	111	462
Bleach caustic extraction stage	460	1916
Remainder of bleaching process	27	113
Total	634	2631

Effects Of Massive Lime Treatment On Effluent Color

Using data from Figures 16 and 19, the maximum amount of effluent that can be treated in a massive lime system (20,000 ppm lime treatment) without exceeding the mill's causticizing requirements is:

$$\frac{(1540 \text{ lb sludge for causticizing per ton unbl pulp})\ (1136 \text{ tons unbl pulp})}{(443{,}666 \text{ lb sludge per } 10^6 \text{ gal. of effluent treated})} = \sim 4 \text{ million gallons}$$

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Assuming the most highly colored effluents would be treated, then all of the bleach caustic extraction stage effluent (2,750,000 gallons per day) and approximately 1,250,000 gallons per day of unbleached decker effluent would be treated for color removal. Combining these two flows would give an influent to the massive lime system having a color of 14,500 APHA units. Based on a color removal of 95% (from Table 7), color of this effluent would be reduced to 725 APHA units after massive lime treatment. This stream would then be combined with mill effluents which had not been treated for color removal. Color of the combined mill effluent would then be 742 APHA units and color loads contributed by various individual sources would be as shown in Table 10.

Table 10

CONTRIBUTION OF EFFLUENT SOURCES TO TOTAL MILL EFFLUENT COLOR WITH MASSIVE LIME TREATMENT OF BLEACH EXTRACTION STAGE AND DECKER EFFLUENTS

Source of Effluent	Color Load (lb color/bl ton)	Contribution to Combined Effluent Color (APHA Color Units)
Pulp mill—general	12	50
Paper mill	5	21
Bleach chlorination stage	111	462
Bleach caustic extraction stage	23	96
Remainder of bleaching process	27	113
Total	178	742

The net effect of the massive lime treatment is a 72% reduction in color of the combined mill effluent (2631 units to 742 units).

Equipment Size

Based on design criteria presented earlier in this report, the massive lime plant for treating four million gallons of effluent per day would require equipment of approximately the following sizes:

- Slaker — Designed to slake 400 tons of reburned lime per day
- Reaction tank — 15 feet in diameter by 15 feet high
- Primary clarifier — 80 feet in diameter by 26 feet high with a center well 25 feet in diameter by 12 feet deep
- Carbonator clarifier — 55 feet in diameter by 24 feet high with a center well (carbonation zone) 35 feet in diameter by 12 feet deep
- Sludge storage tank — 30 feet in diameter by 20 feet high
- Mud filter — 2250 square feet of filter area
- Flue gas (CO_2) blower — To deliver 3000 cubic feet per minute at 5 psig

As described in Section VII, use of the massive lime sludge also results in larger equipment being required in the mill's cooking liquor and recovery processes. Compared with a mill which does not have the massive lime treatment, the following increases in capacity are required.

	Increased Capacity, %
• Green liquor slaker or mixing tank	17.7
• Causticizers and associated equipment	17.7
• White liquor clarifier, storage, and associated equipment	17.7
• Mud washers, storages, and associated equipment	15.8
• Mud filter	6.4
• Lime kiln	6.4
• Pulp washing accessories	5.8
• Evaporators and accessories	5.8
• Recovery boilers and accessories	1.9

Labor Costs For Massive Lime System

With the massive lime treatment system located adjacent to the normal liquor making facilities, operation of the two systems can be controlled by the same personnel. Additional duties imposed by operation of the massive lime system are:

- Observe and inspect all equipment to see that it is performing efficiently.
- Back wash and acid clean the sludge filter. Back washing is required at 6-hour intervals and requires about 20 minutes. Acid cleaning is required once every two days and requires about one hour.
- Test solids concentration once each four hours.
- Test decolored effluent to control loss of lime due to improper carbonation or carbonate settling.
- Observe instrument panelboard for indications of improper operation.
- Keep the area clean.

These additional duties necessitated by operation of the massive lime process should require no more than 8 hours per day for a cost of:

(8 hr) (5.50 $ per hr including fringes, overtime, etc) = $44.00

Maintenance Costs For Massive Lime System

Maintenance costs for the hypothetical mill are estimated to be $50,000 per year ($142.86 per day) more than for a comparable mill operating without the massive lime treatment system.

Electricity Costs For Massive Lime System

The color treatment system requires 1500 connected horsepower. With a load factor of 80%, this is estimated to cost $150 per day.

Steam Costs For Heating Slaker

Approximately 20% (or 0.8 million gallons per day) of the influent must be heated to 210°F for slaking. Some steam is needed to bring the slaker to temperature on startup and some "trim" steam is required for temperature control. Based on an assumed 2°F rise in the slaker, achieved by use of steam, cost of the steam is:

$$\frac{(0.8 \times 10^6 \text{ gal. effluent})\ (8.33 \text{ lb per gal.})\ (2 \text{ BTU per lb})}{(970 \text{ BTU per lb steam})} = 13{,}740 \text{ lb steam per day}$$

$$(13{,}740 \text{ lb steam/day})\ (1.05 \$ \text{ per } 1000 \text{ lb steam}) = \$14.42 \text{ per day}$$

Lime Losses

As indicated in Figures 15 and 17, calcium carbonate losses from the carbonator clarifier are 4445 lb and 727 lb per million gallons for bleach caustic extraction and decker effluents, respectively. A weighted average of these indicates losses of 13,133 lb of calcium carbonate per day. However, when adjusted to compensate for calcium, which is brought into the system with the influents, the net loss is 10,093 lb of calcium carbonate per day. Converting this to tons of new lime (90% calcium oxide) which must be purchased at $25.40 per ton gives a cost for lime of $79.76 per day.

Sodium Losses

Also, as shown in Figures 15 and 17, significant amounts of sodium compounds which enter the system with the reburned lime are lost with the carbonator clarifier effluent. The sodium compounds are essential for cooking and must be replaced by adding sodium sulfate to the mill's chemical recovery system. Cost of the soda lost is:

$$\frac{(10{,}987 \text{ lb } Na_2CO_3 \text{ lost per day})\ (142 \text{ lb } Na_2SO_4)\ (30.40 \$ \text{ per ton } Na_2SO_4)}{(2000 \text{ lb per ton})\qquad(106 \text{ lb } Na_2CO_3)} = \$223.74 \text{ per day}$$

Acid Cleaning Massive Lime Sludge Filter

Eight carboys of inhibited muriatic acid are required every two days for acid cleaning the filter. Cost of this acid is $8.09 per carboy or $32.36 per day. If a bulk system for muriatic acid is installed, costs could be much less.

Increased Operating Costs Of Normal Pulp Mill Operations

The above costs are only the costs directly related to operation of the massive lime treatment plant. As discussed in Section VII, use of the massive lime sludge in the mill's causticizing system affects many of the normal pulp mill operations. Effects which increased costs of

pulp mill operations (based on data from Section VII) are shown in Table 11. Effects which decreased net costs of pulp mill operations (based on data from Sections VI and VII) are shown in Table 12.

Table 11

INCREASED PULP MILL OPERATING COSTS RESULTING FROM MASSIVE LIME TREATMENT

	Effect of Massive Lime Treatment on Pulp Mill Operations	Raw Material or Utility Cost	Increased Pulp Mill Cost ($ per day)
1.	Steam used to heat filter cake and green liquor for causticizing; 313 lb per ton of unbleached pulp produced	$1.05 per 1000 lb of steam	373.35
2.	Added 0.5 lb of defoamer per ton of unbleached pulp to control foaming in the causticizing system	$0.25 per lb of defoamer	141.88
3.	Increased fuel consumption in the lime kiln 158,400 BTU per ton of unbleached pulp	$0.625 per million BTU	112.46
4.	Increased steam used for evaporating black liquor 235 lb of steam per ton of unbleached pulp	$1.05 per 1000 lb of steam	280.31
5.	Increased sodium losses from the system the equivalent of 1.8 lb of Na_2SO_4 per ton of unbleached pulp	$30.40 per ton	31.07
6.	Increased lime losses from the system the equivalent of 1.3 lb of new lime	$25.40 per ton of new lime	18.74

Table 12

PULP MILL SAVINGS OR CREDITS RESULTING FROM MASSIVE LIME TREATMENT

	Effect of Massive Lime Treatment on Pulp Mill Operations	Raw Material or Utility Cost	Savings Realized ($ per day)
1.	Reduced BOD content of the waste water 2695 lb per day	$0.04 per lb BOD	107.79
2.	Increased steam production by 203 lb per ton of unbleached pulp	$1.05 per 1000 lb of steam	242.14
3.	Increased production of electricity by 10.4 KWH per ton of unbleached pulp	$0.007 per KWH	82.70

Capital Costs

Increased capital costs resulting from use of the massive lime color removal process were determined by preparing two capital cost estimates for the hypothetical 1000-tons-per-day mill; one estimate without the color removal process and one including the color removal process. Appendix D gives a comparison of capital cost estimates for the two situations. Including the massive lime process in the hypothetical mill is estimated to increase total mill capital costs $2,704,000.

Annual depreciation, insurance, and taxes, shown in Table 13, were calculated based on the cost estimates given in Appendix D.

Table 13

ANNUAL DEPRECIATION, INSURANCE, AND TAXES

	Without Massive Lime System	With Massive Lime System	Difference
Total investment required	$119,954,000	$122,658,000	$2,704,000
Annual charges for:			
Depreciation	7,254,433	7,422,158	167,725
Insurance	183,287	187,429	4,142
Taxes	1,499,425	1,533,225	33,800
Total	$ 8,937,145	$ 9,142,812	$ 205,667

Assuming the mill operates 350 days per year, the increase in depreciation, insurance, and taxes resulting from use of the massive lime color removal system would be:

$$\frac{(205{,}667\ \$\ \text{per year})}{(350\ \text{days per year})\ (1000\ \text{tons of pulp per day})} = \$0.588\ \text{per ton of pulp}$$

Overall Effects On Costs And Mill Operation

All significant costs incurred or changed as a result of using the massive lime color removal process are summarized in Table 14. These cost data are based on typical values for a *new* mill, and will vary for different situations. Mill size would have a significant effect on the costs. Also, capital investment required for adding a massive lime color removal process to an existing mill would probably be quite different from the costs for including a similar system in a new mill. Some of the reasons for this are; increasing capacity of existing equipment would be more costly than providing the necessary extra capacity in a new installation, space for new equipment may not be readily available, and available space may be in relatively unfavorable locations in existing mills.

Costs shown in Table 14 are for a hypothetical mill producing 1000 tons of bleached kraft pulp which is 75% pine and 25% hardwood. Using all of the mill's available lime allowed 4,000,000 gallons of the mill's 29,000,000 gallons of effluent to be treated for color removal. This level of treatment removed approximately 72% of the mill's total effluent color, giving a final combined mill effluent color of 742 APHA color units. These benefits were achieved in the hypothetical mill at a cost of $1.80 per ton of bleached pulp. This includes costs of depreciation, taxes, and insurance for the capital expenditures required as a result of massive lime treatment.

Table 14

Summary of Effects of Massive Lime System on Operating Costs for Hypothetical Mill Producing 1000 Tons of Bleached Pulp Per Day

	— Costs of Massive Lime System —			
	($ per day)		$ per ton of bleached pulp	
Costs of operating massive lime system,				
Operating labor	44.00		0.044	
Maintenance	142.86		0.143	
Electricity	150.00		0.150	
Steam	14.43		0.014	
Lime	79.76		0.080	
Saltcake (Na_2SO_4)	223.74		0.224	
Muriatic acid	32.36		0.032	
		687.15		0.687
Increased pulp mill operating costs resulting from use of massive lime process,				
Steam to heat filter cake and green liquor	373.35		0.373	
Defoamer	141.88		0.142	
Increased fuel to lime kiln	112.46		0.112	
Increased steam for black liquor evaporation	280.31		0.280	
Sodium losses	31.07		0.031	
Lime losses	18.74		0.019	
		957.81		0.958
Credits resulting from massive lime treatment				
Reduced BOD load	107.79		0.108	
Increased steam production	242.14		0.242	
Increased production of electricity	82.70		0.083	
		(432.63)		(0.433)
Annual depreciation, insurance, and taxes		587.62		0.588
Total		$1799.95		$1.800

SECTION X

ACKNOWLEDGMENTS

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Suggestions for the application and design for equipment used in carbonating liquids, by Mr. Charles L. Davis, Jr., Interstate Paper Company, Riceboro, Georgia, and Mr. Ed Spruill, Continental Can Corporation, Hodge, Louisiana, were useful in overcoming deficiencies in performance of original design.

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SECTION XI

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SECTION XII

GLOSSARY

Active Cooking Chemical--	Sodium hydroxide (NaOH) and sodium sulfide (Na_2S) in kraft pulp mill liquor; usually expressed as the equivalent amount of Na_2O; also called active alkali.
Batch Digester–	A large pressure vessel in which a batch of wood chips is cooked with white liquor to produce pulp; the digester is filled, capped, brought to temperature and pressure, held at these conditions for some period then emptied, a batch cooking process.
Black Liquor–	Spent cooking liquor from the kraft pulping process, which contains spent cooking chemicals plus materials from the wood (mostly lignin) which reacted with white liquor during the cooking process and dissolved.
Black Liquor Solids–	The residue left when evaporating a sample of black liquor to dryness.
BOD–	Biochemical oxygen demand.
Brown Stock Washing–	A countercurrent multistage pulp washing process immediately after cooking; used to remove (wash) from the pulp the spent cooking chemicals and materials made soluble during the cook.
Ca-Organics–	Those organic compounds which have combined with calcium to form insoluble compounds.
Clarifier–	Equipment for removing settleable solids from liquids.
Causticizing Power–	Generally expressed as a percent causticizing value indicating the percent of a sample of lime which when slaked will react with Na_2CO_3 to produce NaOH.
COD–	Chemical oxygen demand.
Continuous Digester–	A large pressure vessel for cooking wood chips with white liquor in a manner that allows wood chips (and white liquor) to be added continuously and pulp to be removed continuously.
Consistency–	Ovendry weight of pulp in a pulp-water slurry divided by the total slurry weight; usually expressed as a percentage.

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Cooking Liquor– Liquor which is added to the digester to provide the necessary chemicals for the cooking process; white liquor.

Digester– A large pressure vessel for cooking wood chips (with white liquor) to produce pulp.

Dregs– Heavy solids that settle in the slaker and are removed from the process.

Green Liquor– A solution of recovered pulp mill cooking chemicals, formed by dissolving recovery furnace smelt in water; primarily sodium carbonate, sodium sulfide, and water.

Launder– A trough used to collect and transport liquid.

Pulp Yield– The percentage obtained by dividing the ovendry weight of pulp produced by the ovendry weight of wood used to produce that amount of pulp.

Reburned Lime– Lime produced from recycled calcium carbonate which is produced in the causticizing process and removed in the white liquor clarifier.

Recausticizing– Process of reacting green liquor with slaked lime to produce white liquor; Na_2CO_3 in the green liquor reacts with $Ca(OH)_2$ to produce NaOH.

Recovery Furnace– The chemical recovery unit in which concentrated black liquor is burned; heat from this combustion is used to generate steam; sulfur compounds are reduced to sulfides; inorganic sodium and sulfur compounds are recovered from the bottom of the unit as a smelt which is dissolved in water to form green liquor.

Slaker– Equipment in which the slaking process occurs.

Slaking– Process of adding lime to water to convert the calcium oxide to calcium hydroxide.

Soda– Generally used in the paper industry to refer to sodium compounds present in pulp mill liquors.

Strong Black Liquor– Black liquor which has been increased to approximately 50% black liquor solids or more by evaporation of water from weak black liquor.

Weak Black Liquor– The dilute (approximately 10 to 15% solids) black liquor, before evaporation; obtained from the brown stock washing operation.

White Liquor– Highly alkaline chemical solution used to digest (cook) wood in the kraft (sulfate) pulping process; formed by the reaction of green liquor with slaked lime; active chemicals are primarily NaOH and Na_2S.

SECTION XIII

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APPENDIX A

CHRONOLOGICAL LIST OF MODIFICATIONS MADE TO MASSIVE LIME DEMONSTRATION PLANT

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CHRONOLOGICAL LIST OF MODIFICATIONS MADE TO MASSIVE LIME DEMONSTRATION PLANT

	DATE	CHANGE	REASON FOR CHANGE
1.	3/1/70	Started putting flow of mud from carbonator clarifier into the mill's mud washer instead of into the unfiltered mud tank.	Calcium hydroxide carryover from the carbonator clarifier caused the mill's mud filter to glaze over.
2.	3/6/70	Changed the flow of carbonator clarifier mud again; this time put it into the massive lime system sludge storage tank.	The change made on 3/1/70 caused a problem in settling of the mill's regular calcium carbonate mud, and a foaming problem which resulted in turbid wash liquor.
3.	4/15/70	Filtrate from the sludge filter was put into the primary clarifier instead of into the carbonator clarifier.	A significant quantity of solids were going through the sludge filter and being carried with the filtrate.
4.	4/28/70	Started putting filtrate from the sludge filter into the reaction tank.	The change made on 4/15/70 resulted in considerably impaired settling rates in the primary clarifier.
5.	5/11/70	Started putting filtrate from the sludge filter into the massive lime system slaker. This also required increased heat (steam) input into the slaker.	Settling rate remained impaired.
6.	5/12/70	Installed new flue gas distribution system in the carbonator clarifier.	Scaling problems with original system made it unsatisfactory.
7.	6/8/70	Extended center well of primary clarifier to a depth of 10 feet below the liquid level (it originally extended only two feet below liquid level).	To try to eliminate excessive carryover of suspended solids from the primary clarifier.

	DATE	CHANGE	REASON FOR CHANGE
8.	7/6/70	Removed four feet of the eight-foot extension added to the primary clarifier center well 6/8/70.	Excessive carryover continued after center well had been extended on 6/8/70.
9.	9/10/70	Enlarged and levelled overflow weirs in primary clarifier and in carbonator clarifier.	To achieve a more even distribution of overflow from the weirs and hopefully less carryover of suspended solids.
10.	9/10/70	Changed flue gas flow control from back pressure control to atmospheric relief control	Excessive back pressure was causing electrical overloads on the flue gas blower.
11.	9/10/70	Relocated pH control of the carbonator so pH of the center well could be measured almost immediately.	Original pH measurement of the clarifier overflow gave a 3-hour lag in measurement so response to changes was too slow and control was not good with the original system.
12.	9/10/70	The pipe transferring liquid from the primary clarifier to the carbonator clarifier was replaced with an open trough.	Scaling problems were encountered with the pipe and a trough should be easier to clean.
13.	10/16/70	Installed experimental clarifier in parallel with primary clarifier.	To provide a small-scale means of studying way to eliminate the foaming and suspended solids carryover problems of the primary clarifier. To evaluate new launder and center well design.
14.	10/22/70	Tried adding high molecular weight polymer settling aids.	To see if carryover of suspended solids could be eliminated.
15.	1/14/71	Installed conical center well, radial launders, and Yarway foam suppressing showers in the primary clarifier.	To eliminate foaming and carryover problems of the primary clarifier.
16.	1/14/71	Replaced the original flue gas blower with a Nash pump.	Severe maintenance problems had been experienced with the original type of blower.

	DATE	CHANGE	REASON FOR CHANGE
17.	1/14/71	Changed flow of filtrate from sludge filter so it again went to the reaction tank.	With the flow going into the slaker, steam consumption in the slaker was high.
18.	7/9/71	Changed sludge filter from precoat operation to continuous belt operation.	To compare the two methods of operations.

APPENDIX B

DESCRIPTION OF ANALYTICAL TESTING TECHNIQUES USED

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Tests On Liquid Effluents

1. Procedures for the following tests which were used during this project are described in the book, *Standard Methods For The Examination Of Water And Wastewater,* Twelfth Edition, published by American Public Health Association, Inc., New York, October 1967.

Test	Page Numbers
APHA color[1]	127 - 129
Total Solids Content	244 - 245
Suspended Solids Content	245 - 246
Dissolved Solids Content	246 - 247
Biochemical Oxygen Demand (BOD_5)	415 - 421
Chemical Oxygen Demand (COD)	510 - 514

[1]A Klett-Summerson colorimeter Model 900-3 was used for color comparison instead of Nessler tubes.

2. Procedures used to determine total carbon (TC), total organic carbon (TOC), and total inorganic carbon present in the effluents are described in the FWPCA manual *Methods for Chemical Analysis of Water and Wastes,* U. S. Dept. of Interior, November 1969, p 211.

3. Laboratory Massive Lime Treatment

   a. Collect a 3-liter sample of effluent.

   b. To a 600-ml portion of the sample add 60 grams of lime (20,000 ppm) and heat with stirring until the lime is slaked.

   c. Add the 600-ml portion to the remainder of the 3-liter sample while stirring at maximum rpm with a Phipps and Bird variable speed stirrer.

   d. After 2 minutes at high speed, reduce stirrer speed to 50 rpm for 5 minutes, then stop the stirrer and allow the sludge to settle.

   e. Start time when the stirrer is cut-off.

   f. Record sediment height at 5, 10, 15, 20, and 30 minutes.

   g. Remove supernatant liquid for carbonation (step j) and test as desired.

   h. Filter the residue through Whatman No. 41 filter paper on a Buchner funnel to collect the settled sludge.

   i. Dry the collected sludge in an oven at 105°C, weigh, and save for testing as desired.

   j. Bubble CO_2 through sample (from step g) of supernatant liquid until pH 11.0 is reached.

   k. Filter carbonated sample through Whatman No. 41 filter paper on a Buchner funnel.

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l. Save filtrate for testing as desired.

m. Dry calcium carbonate mud in oven at 105°C, weigh, and save for testing as desired.

4. Sodium Content

Sodium was determined using a Beckman DU-2 flame spectrophotometer.

5. Calcium Content

a. Pipette a 25-ml sample into a crucible and acidify with HCl.

b. Evaporate to dryness.

c. Place in muffle furnace at 600°F for 10 to 15 minutes.

d. Cool, then dissolve residue in concentrated HCl.

e. Place HCl solution in muffle furnace at 600°F for 10 to 15 minutes.

f. Cool, then dissolve residue in 6N HCl.

g. Evaporate to dryness, then place in muffle furnace at 600°F for 10 to 15 minutes.

h. Repeat steps f and g.

i. Dissolve the residue in hot distilled water.

j. Filter the solution through Whatman No. 41 filter paper into a 100-ml volumetric flask and make to the mark.

k. To a 25-ml sample of the filtrate add calcium indicator powder and 6 to 8 drops of calcium buffer.

l. Titrate with 0.001M EDTA.

m. Calculate Ca concentration by $\% \text{ Ca} = \frac{\text{ml titration} \times 0.0160}{25}$

6. Chlorides Concentration

a. Pipette a suitable volume of liquid into an Erlenmeyer flask.

b. Neutralize with dilute H_2SO_4 or NaOH solution.

c. Add 1 ml of 2% potassium chromate solution.

d. Titrate with 0.0142N $AgNO_3$ to the first color change.

e. Calculate chlorides concentration by;

$$\text{ppm chlorides} = \frac{\text{ml titration} \times 498}{\text{ml of sample}}$$

Tests On Pulp Mill Liquors

1. Analysis of Cooking Chemicals in Green Liquor And White Liquor

   To determine total sodium compounds (total alkali) which are in an active form ($NaOH$ or Na_2S) or which can be converted (Na_2CO_3) to active cooking chemicals;

   a. Pipette 5 ml of the liquor sample into a 250-ml Erlenmeyer flask.

   b. Add approximately 100 ml of distilled water and 2 or 3 drops of Methyl Orange indicator.

   c. Titrate to the Methyl Orange end point with 1N HCl.

   d. Record the ml of acid used in the titration as the value, A.

   e. Calculate total alkali as the Na_2O equivalent by;

      Total Alkali, as gpl $Na_2O = 6.20\ A$

   To determine the NaOH and Na_2S contents;

   a. Pipette a 25-ml sample of the liquor into a 250-ml volumetric flask.

   b. Fill to the mark with 10% $BaCl_2$, shake, and let settle for 15 minutes.

   c. Pipette 50 ml of the supernatant liquid into a 250-ml Erlenmeyer flask and titrate to the phenolphathalein end point.

   d. Record ml of 1N HCl used in this titration as the value, B.

   e. Continue to titrate with 1N HCl to the Methyl Orange end point.

   f. Record the total ml of 1N HCl used in titrating to both end points as the value, C.

   g. Calculate NaOH and Na_2S content as the Na_2O equivalent by;

      NaOH, as gpl $Na_2O = 6.2\ [B - (C - B)]$

      Na_2S, as gpl $Na_2O = 12.4\ (C - B)$

      Na_2CO_3, as gpl $Na_2O = 6.2\ (A - C)$

2. Unreduced Na_2SO_4

   a. Pipette 10 ml of liquor into a small beaker.

   b. Dilute with distilled water and add 10 ml of concentrated HCl.

   c. Boil on a hot plate until the solution is clear, then filter through qualitative filter paper.

   d. Add 10 ml of 12% $BaCl_2$ to the filtrate and let stand on hot plate without boiling until precipitate has settled.

   e. Filter through a weighed Gooch crucible containing a suitable filter pad to retain the precipitate.

f. Wash, dry, and weigh the crucible.

g. Calculate unreduced Na_2SO_4 by:

$$\text{Unreduced } Na_2SO_4, \text{ as gpl } Na_2O = 26.61 \text{ (wt of precipitate)}$$

3. Settling Time For Clarifier Sludge

   a. Collect representative sample.

   b. Agitate sample to get uniform slurry.

   c. Fill 3-liter beaker and start timing.

   d. Measure and record temperature.

   e. Record height of sediment at 5, 10, 15, 20, and 30 minutes.

4. Tests for sodium content, calcium content, chlorides concentration, and COD were made using the procedures described for testing Liquid Effluents.

Tests On New And Reburned Lime

1. Available Lime

   a. Weigh 1 gram lime and add approximately 25 ml water.

   b. Boil 5 minutes.

   c. Cool, add 40 grams sugar, stopper, and shake every 15 minutes for 2 hours.

   d. Filter through Whatman No. 42 filter paper on a Buchner funnel and wash with 100 ml of 10% sugar solution.

   e. Titrate filtrate with 1N HCl to a phenolphathalein end point.

   f. Calculate:

      % Available Lime = 2.804 (ml Titration)

2. HCl Insolubles In Lime And Lime Mud

   a. Weigh a 25-gram sample into a beaker.

   b. Bring volume to approximately 500 ml with distilled water.

   c. Slowly add an excess of concentrated HCl and bring to boil.

   d. Filter through a weighed Gooch crucible containing an asbestos filter pad, wash with hot water, dry, and reweigh.

   e. Save filtrate for sodium, analysis which can be run using procedure as described for testing Liquid Effluents.

   f. Calculate:

$$\text{Wt \% Insolubles} = \frac{\text{Wt Residue} \times 100}{\text{Wt of sample}}$$

3. Causticizing Value

   a. Weigh 212 grams of soda ash and 112 grams of lime.

   b. Heat 1800 ml of distilled water to approximately 160° F in a 2-liter beaker.

   c. Slowly add the soda ash and lime from step (a.) to the heated water.

   d. Boil contents for 1 hour.

   e. Remove the container from the hot plate and allow precipitate to settle to a height of 2 inches.

   f. Pipette 5 ml of supernatant liquid into an Erlenmeyer flask.

   g. Titrate with 1N HCl to phenolphthalein and Methyl Orange end points recording ml of acid to titrate to the first end point as P and that to the second as M.

h. Calculate:

$$\% \text{ Causticizing Value} = \frac{M - 2(M - P)}{0.01\ M}$$

4. Tests for sodium content were made on liquid from 3 above using procedures described previously in tests for Pulp Mill Liquors.

APPENDIX C

OPERATIONAL TEST DATA

Table C-1

100% BLEACH PLANT CAUSTIC EXTRACTION STAGE EFFLUENT

FEED TO MASSIVE LIME PROCESS FEBRUARY 8 – MARCH 10, 1971

Date, 1971	Flow Rate, gpm	APHA Color Units	Suspended Solids, ppm	Dissolved Solids, ppm	Total Carbon, ppm	Inorganic Carbon, ppm	Organic Carbon, ppm	Ca, ppm	Cl, ppm	pH	BOD_5, ppm
2/08	200	15,300	40	5180	1125	50	1075	46	1268	10.9	–
2/09	100	16,500	10	4850	1350	100	1250	31	1145	11.0	336
2/10	100	18,150	20	4820	1475	100	1375	37	1155	9.8	–
2/12	250	18,150	10	4790	1450	50	1400	57	1145	10.3	–
2/13	250	18,150	50	4580	1450	75	1375	67	1035	10.0	–
2/14	150	16,500	40	4630	1125	75	1050	34	1175	9.8	–
2/15	150	23,600	40	6070	1750	50	1700	48	1604	9.8	342
2/16	150	16,500	60	5030	1250	75	1175	37	1254	9.8	–
2/17	150	23,600	0	5870	1725	50	1675	56	1530	10.0	–
2/18	150	23,200	20	6210	1550	75	1475	60	1520	10.0	348
2/19	150	23,200	30	6080	1525	75	1450	40	1510	10.3	–
2/20	150	19,050	20	4840	1225	50	1175	24	1308	9.9	–
2/21	200	21,400	20	4220	1425	75	1350	29	1409	8.6	–
2/22	200	28,250	20	7060	1750	125	1625	42	1782	10.4	–
2/23	150	29,000	30	7220	1750	100	1650	48	1892	9.8	–
2/24	150	26,750	30	7900	1925	75	1850	58	2051	10.3	444
2/27	150	24,200	10	6710	1725	75	1650	40	1820	10.2	–
2/28	150	22,600	10	5940	1375	75	1300	50	1693	10.2	–
3/01	150	22,000	30	6370	1500	100	1400	41	1594	10.2	–
3/02	200	20,000	20	5590	1450	100	1350	38	1534	10.4	288
3/03	225	24,800	30	–	1700	25	1675	69	–	9.8	–
3/04	200	21,000	20	–	1475	75	1400	52	–	10.4	–
3/05	200	20,400	20	–	1475	75	1400	35	–	10.1	–
3/06	175	24,200	10	–	1700	100	1600	30	–	10.1	–
3/07	175	23,600	40	–	1600	100	1500	43	–	9.4	–
3/08	175	23,600	30	–	1700	75	1625	36	–	9.9	360
3/09	150	21,000	50	–	1600	25	1575	30	–	9.8	–
3/10	150	18,600	30	–	1400	75	1375	39	–	9.7	–

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Table C-2

50% BLEACH PLANT CAUSTIC EXTRACTION STAGE EFFLUENT AND 50% UNBLEACHED KRAFT DECKER EFFLUENT

FEED TO MASSIVE LIME PROCESS APRIL 7 – MAY 15, 1971

Date, 1971	Flow Rate, gpm	APHA Color Units	Suspended Solids, ppm	Dissolved Solids, ppm	Total Carbon, ppm	Inorganic Carbon, ppm	Organic Carbon, ppm	Ca, ppm	Cl, ppm	pH	BOD_5, ppm
4/07	400	10,200	50	2930	900	75	825	18	594	10.2	–
4/08	400	8000	70	2940	875	50	825	30	553	10.5	–
4/09	450	16,500	30	3260	1000	25	975	21	577	11.4	–
4/10	450	17,500	100	2990	925	25	925	27	557	10.5	294
4/11	500	8300	50	2490	825	25	800	28	508	10.0	–
4/12	500	8900	70	3150	775	25	750	34	598	10.2	–
4/13	500	11,700	70	3370	925	50	875	47	815	9.4	–
4/14	400	9300	20	3050	875	50	825	29	687	10.3	270
4/15	500	8300	60	2750	800	25	775	39	674	10.1	–
4/16	450	8600	70	3030	950	75	875	23	704	10.3	–
4/17	450	12,900	50	3760	1000	75	925	27	–	9.9	–
4/18	400	12,900	70	3800	1075	50	1025	24	–	10.2	–
4/19	300	12,700	60	3710	1025	75	950	23	–	9.6	–
4/20	300	10,200	10	3720	1025	50	975	36	–	10.8	–
5/01	300	7300	10	2900	750	75	675	25	594	10.8	–
5/02	300	5350	10	2220	650	50	600	27	463	10.2	–
5/03	300	8300	0	2980	825	50	775	24	604	10.8	–
5/04	400	10,500	10	3480	1225	75	1150	25	634	10.4	390
5/05	400	9400	20	2920	775	25	750	28	684	10.6	–
5/06	450	8900	40	2920	700	50	650	26	583	10.2	–
5/07	450	9100	30	2920	825	25	800	29	583	9.8	–
5/08	450	10,200	0	2920	875	50	825	27	583	9.9	–
5/09	350	10,050	20	2920	900	75	825	38	583	10.0	–
5/10	350	10,950	30	2920	725	75	650	29	583	9.7	–
5/11	350	9300	40	2920	750	100	650	31	583	9.8	–
5/12	400	10,500	30	2920	750	75	675	44	583	10.0	246
5/13	400	10,050	40	2920	750	75	675	42	583	9.8	–
5/14	400	8600	50	2920	650	50	600	38	583	9.8	–
5/15	300	6750	70	2920	550	25	525	29	583	10.2	–

Table C-3

100% UNBLEACHED KRAFT DECKER EFFLUENT

FEED TO MASSIVE LIME PROCESS MAY 29 – JULY 2, 1971

Date, 1971	Flow Rate, gpm	APHA Color Units	Suspended Solids, ppm	Dissolved Solids, ppm	Total Carbon, ppm	Inorganic Carbon, ppm	Organic Carbon, ppm	Na, ppm	Ca, ppm	Cl, ppm	pH	BOD_5, ppm
5/29	525	1020	60	1190	275	25	250	370	12		10.0	–
5/30	525	860	140	960	200	25	175	320	24	65	9.7	–
5/31	525	730	50	970	230	20	210	250	18	65	9.8	–
6/01	525	700	40	780	170	20	150	260	17	60	9.3	–
6/02	525	605	70	610	160	10	150	310	27	75	9.2	114
6/03	525	525	50	680	180	10	170	260	15	79	9.3	–
6/04	540	470	120	870	160	10	150	230	29	85	9.2	–
6/05	540	675	60	1040	130	10	120	185	19	100	9.2	–
6/06	540	800	130	990	320	0	320	270	23	110	9.3	138
6/07	540	890	120	1120	110	10	100	300	22	79	10.5	–
6/08	540	1270	60	1490	350	20	330	300	19	50	10.9	–
6/10	540	730	50	1030	260	10	250	246	14	50	10.0	150
6/15	530	970	120	1040	350	10	340	250	21	50	10.2	150
6/16	550	2360	20	1340	740	20	720	525	18	40	10.8	–
6/17	550	970	60	1200	350	20	330	233	26	35	10.0	–
6/18	550	960	200	1040	340	10	330	340	33	59	10.4	–
6/19	550	885	710	1490	310	10	300	265	26	50	9.2	–
6/20	550	1125	170	1200	350	30	320	255	29	50	9.0	–
6/21	550	830	0	1040	270	10	260	235	17	95	9.4	–
6/22	550	590	100	1180	180	10	170	190	36	–	9.8	–
6/23	550	470	120	1470	230	10	220	160	30	90	9.0	96
6/24	550	455	100	1250	160	10	150	178	23	85	9.4	126
6/25	550	910	50	1230	340	20	320	320	12	40	10.1	–
6/26	550	940	40	1280	350	20	330	305	16	40	10.1	–
6/28	550	1100	40	1180	400	20	380	305	12	221	9.7	–
6/29	550	1320	30	1300	430	10	420	275	19	79	9.5	–
6/30	550	660	30	880	290	10	280	240	24	74	9.0	–
7/01	550	1100	60	1220	–	–	–	300	22	45	9.5	192
7/02	550	1180	20	770	–	–	–	360	33	35	10.0	–

Table C-4

EFFLUENT FROM PRIMARY CLARIFIER WHILE TREATING 100% BLEACH PLANT CAUSTIC EXTRACTION STAGE EFFLUENT

FEBRUARY 8 – MARCH 10, 1971

Date, 1971	APHA Color Units	Suspended Solids, ppm	Ca, ppm
2/08	1530	130	472
2/09	1270	30	492
2/10	1270	30	330
2/12	1365	380	403
2/13	1455	–	–
2/14	1245	–	–
2/15	1455	–	–
2/16	1095	–	–
2/17	1245	–	–
2/18	1365	880	917
2/19	1605	810	896
2/20	1410	40	387
2/21	1815	60	334
2/22	1695	–	–
2/23	1770	460	757
2/24	1815	30	260
2/27	1455	50	366
2/28	1500	30	346
3/01	1170	50	398
3/02	1365	20	396
3/03	1605	60	396
3/04	1530	–	–
3/05	1320	220	592
3/06	1410	200	424
3/07	1500	100	512
3/08	1650	–	–
3/09	1695	80	400
3/10	1240	140	592

Table C-5

EFFLUENT FROM PRIMARY CLARIFIER WHILE TREATING 50% BLEACH PLANT CAUSTIC EXTRACTION STAGE EFFLUENT AND 50% UNBLEACHED KRAFT DECKER EFFLUENT

APRIL 7 – MAY 15, 1971

Date, 1971	APHA Color Units	Suspended Solids, ppm	Ca, ppm
4/07	365	42[illegible]	107
4/08	400	20	492
4/09	–	120	240
4/10	356	80	422
4/11	–	–	–
4/12	–	–	–
4/13	510	–	–
4/14	445	0	380
4/15	400	30	384
4/16	500	100	572
4/17	780	–	–
4/18	1070	–	–
4/19	1020	–	–
4/20	675	10	479
5/01	970	30	432
5/02	240	30	454
5/03	350	20	468
5/04	620	20	468
5/05	700	20	447
5/06	390	40	513
5/07	565	–	–
5/08	390	100	750
5/09	430	40	630
5/10	415	30	514
5/11	455	60	504
5/12	455	80	570
5/13	525	–	–
5/14	320	140	459
5/15	230	130	553

Table C-6

EFFLUENT FROM PRIMARY CLARIFIER WHILE TREATING 100% UNBLEACHED KRAFT DECKER EFFLUENT

MAY 29 – JULY 2, 1971

Date, 1971	APHA Color Units	Suspended Solids, ppm	Ca, ppm
5/29	185	10	461
5/30	230	10	514
5/31	135	10	537
6/01	175	10	497
6/02	200	20	497
6/03	160	10	510
6/04	160	70	439
6/05	160	20	506
6/06	185	80	496
6/07	215	160	491
6/08	220	60	424
6/10	180	200	437
6/15	230	40	488
6/16	285	10	497
6/17	240	10	495
6/18	255	20	516
6/19	230	130	515
6/20	255	130	550
6/21	230	220	534
6/22	230	75	510
6/23	230	390	535
6/24	230	200	565
6/25	240	50	494
6/26	295	10	530
6/28	270	0	558
6/29	255	10	565
6/30	255	10	597
7/01	295	10	522
7/02	285	0	538

Table C-7

DECOLORED EFFLUENT FROM CARBONATOR CLARIFIER WHILE TREATING 100% BLEACH PLANT CAUSTIC EXTRACTION STAGE EFFLUENT

FEBRUARY 8 – MARCH 10, 1971

Date, 1971	APHA Color Units	Suspended Solids, ppm	Dissolved Solids, ppm	Total Carbon, ppm	Inorganic Carbon, ppm	Organic Carbon, ppm	Total Ca, ppm	Soluble Ca, ppm	Cl, ppm	pH	BOD_5, ppm
2/08	1350	210	5240	380	20	360	66	36	1248	12.6	–
2/09	1070	10	5230	360	20	340	–	–	1096	12.8	198
2/10	1240	420	4240	480	130	350	215	30	1095	12.4	–
2/12	1290	30	5260	380	10	370	–	–	1085	12.9	–
2/13	940	–	4900	390	30	360	–	–	1095	13.2	–
2/14	640	370	4540	310	50	260	179	159	1135	12.7	–
2/15	565	150	5750	290	10	280	–	–	1355	13.0	312
2/16	1020	–	3860	500	160	340	–	–	1215	12.4	–
2/17	1125	20	5160	370	0	370	–	–	1479	12.9	–
2/18	1245	230	5380	350	0	350	–	–	1530	12.9	258
2/19	1320	370	4880	460	60	400	132	84	1530	12.6	–
2/20	1365	140	5830	340	20	320	309	260	1449	12.6	–
2/21	1650	–	4400	500	130	370	264	83	1308	11.7	–
2/22	1050	200	6100	390	20	370	–	–	1711	12.4	–
2/23	1180	260	6910	420	30	390	242	166	1793	12.3	–
2/24	1605	90	6080	460	10	450	166	127	1872	12.3	342
2/27	1530	170	6670	440	30	410	–	–	1780	12.8	–
2/28	1605	340	6500	380	20	360	192	168	1600	12.8	–
3/01	1245	140	6100	380	20	360	212	176	1594	12.8	–
3/02	1815	–	5140	650	150	500	256	156	1594	12.2	246
3/03	2000	–	–	660	220	440	260	96	1474	11.9	–
3/04	655	170	–	350	20	330	–	–	–	12.7	–
3/05	1280	–	–	680	150	530	322	58	–	12.5	–
3/06	1357	–	–	590	190	400	228	46	–	11.8	–
3/07	1695	–	–	650	230	420	298	52	–	11.9	–
3/08	750	190	–	350	10	340	–	–	–	12.9	227
3/09	1695	–	–	610	250	360	230	46	–	12.1	–
3/10	1130	–	–	420	110	310	212	44	–	12.3	–

Table C-8

DECOLORED EFFLUENT FROM CARBONATOR CLARIFIER WHILE TREATING 50% BLEACH PLANT CAUSTIC EXTRACTION STAGE EFFLUENT AND 50% UNBLEACHED KRAFT DECKER EFFLUENT

APRIL 7 – MAY 15, 1971

Date, 1971	APHA Color Units	Suspended Solids, ppm	Dissolved Solids, ppm	Total Carbon, ppm	Inorganic Carbon, ppm	Organic Carbon, ppm	Total Ca, ppm	Soluble Ca, ppm	Cl, ppm	pH	BOD_5, ppm
4/07	415	290	3170	250	40	210	100	22	594	12.3	–
4/08	470	–	2850	350	90	260	202	21	594	12.1	–
4/09	800	–	2760	460	60	400	–	–	577	12.6	–
4/10	428	–	2440	450	50	400	212	33	557	12.7	252
4/11	400	–	2850	290	50	240	216	94	518	12.3	–
4/12	390	150	4360	200	10	190	184	156	598	12.7	–
4/14	445	210	3670	280	20	260	116	74	637	12.5	276
4/15	400	240	3790	220	20	200	132	60	–	12.6	–
4/16	605	–	3400	550	140	410	246	30	765	12.0	–
4/17	445	40	3880	220	30	190	–	–	–	12.9	–
4/18	415	60	4420	260	20	240	–	–	–	12.8	–
4/19	470	60	3960	290	30	260	–	–	–	12.8	–
4/20	700	–	3060	400	120	280	213	28	–	12.2	–
5/01	–	–	–	–	–	–	–	–	–	–	–
5/02	335	220	2760	360	110	250	109	24	55	12.3	–
5/03	445	300	2860	380	130	250	124	40	594	11.4	–
5/04	635	–	2820	620	150	470	280	21	604	11.6	186
5/05	675	–	3000	380	110	270	134	41	684	12.0	210
5/06	365	210	3000	180	20	160	128	57	614	12.2	–
5/07	295	100	3000	310	10	300	–	–	614	12.3	–
5/08	455	–	3000	290	80	210	245	69	614	11.5	–
5/09	455	–	3000	290	90	200	249	83	614	11.5	–
5/10	535	510	3000	360	100	260	423	115	614	11.2	–
5/11	510	–	3000	250	90	160	221	130	614	11.4	–
5/12	470	–	3000	270	120	150	250	142	614	11.6	192
5/13	295	130	3000	140	30	110	–	–	614	12.5	–
5/14	295	410	3000	240	40	200	384	160	614	12.2	–
5/15	185	130	3000	190	10	180	87	67	614	12.1	–

Table C-9

DECOLORED EFFLUENT FROM CARBONATOR CLARIFIER WHILE TREATING 100% UNBLEACHED KRAFT DECKER EFFLUENT

MAY 29 -- JULY 2, 1971

Date, 1971	APHA Color Units	Suspended Solids, ppm	Dissolved Solids, ppm	Total Carbon, ppm	Inorganic Carbon, ppm	Organic Carbon, ppm	Na, ppm	Total Ca, ppm	Soluble Ca, ppm	Cl, ppm	pH	BOD_5, ppm
5/29	255	65	--	220	70	150	625	44	22	--	11.4	--
5/30	255	20	1620	200	60	140	525	50	43	65	11.6	--
5/31	175	85	1540	140	30	110	500	44	15	75	11.4	--
6/01	175	110	1640	170	30	140	510	52	17	75	11.7	--
6/02	200	60	1600	100	20	80	560	71	50	90	12.4	90
6/03	160	150	1340	90	10	80	514	73	20	104	11.8	--
6/04	160	60	1390	70	10	60	520	31	7	85	12.5	--
6/05	185	100	1640	40	10	--	455	99	63	100	12.5	--
6/06	215	115	1520	215	10	215	533	59	21	110	12.1	78
6/07	215	60	1620	140	10	130	560	31	13	79	12.4	--
6/08	220	65	1430	190	40	150	655	33	14	45	12.3	--
6/10	160	20	1780	140	30	110	608	25	20	55	11.8	65
6/15	230	35	1920	180	30	150	540	24	12	89	12.7	76
6/16	285	90	1990	250	10	240	645	109	65	35	11.9	--
6/17	240	60	1950	200	30	170	475	98	82	60	11.8	--
6/18	248	10	1850	100	30	70	570	83	74	89	11.5	--
6/19	240	150	1200	120	10	110	480	85	42	75	11.3	--
6/20	285	25	1750	160	10	150	475	90	82	75	11.2	--
6/21	255	110	1460	80	10	70	465	146	73	99	11.5	--
6/22	240	90	1590	90	10	80	415	119	85	75	12.0	--
6/23	240	70	1530	80	10	70	530	130	103	90	12.1	50
6/24	240	100	1570	90	20	70	362	210	174	85	12.4	52
6/25	240	70	1780	140	10	130	675	93	66	75	12.1	--
6/26[1]	285	0	2870	220	10	210	570	504	502	50	12.7	--
6/28[1]	255	30	2400	180	10	170	540	516	502	50	12.7	--
6/29[1]	255	10	2560	150	0	150	475	536	534	75	12.7	--
6/30[1]	255	20	2250	190	10	180	415	558	546	74	12.8	--
7/01[1]	270	10	2540	--	--	--	500	494	486	45	12.5	117
7/02[1]	335	60	2270	--	--	--	600	474	458	35	12.0	--

[1] Effluent was not carbonated during this period.

Table C-10

GREEN LIQUOR ANALYSES

Period		Sodium Compounds, gpl as Na_2O				Unreduced Na_2SO_4 as Na_2O, gpl	APHA Color, Units	Ca, ppm	Cl, ppm	COD, ppm	Carbon, ppm		
		Na_2CO_3	NaOH	Na_2S	Total						Inorganic	Organic	Total
6/15/69 - 8/11/69	Av	–	–	–	–	2.65	–	11	–	32,242	–	–	–
	Max.	–	–	–	–	3.57	–	20	–	38,512	–	–	–
	Min	–	–	–	–	1.82	–	10	–	27,480	–	–	–
11/3/69 - 1/20/70	Av	75.6	21.1	34.7	131.4	3.30	–	8	818	33,907	10,400	4200	14,600
	Max.	83.7	26.0	37.2	137.1	4.60	–	13	906	38,520	–	–	–
	Min	67.5	13.6	29.1	128.4	2.47	–	5	654	29,540	–	–	–
4/20/70 - 6/2/70	Av	76.5	23.0	34.4	133.9	3.48	–	16	940	31,237	9394	2360	11,753
	Max.	82.0	28.5	37.8	139.5	5.22	–	33	1273	36,723	11,492	3910	12,775
	Min	67.5	17.4	31.0	130.8	2.16	–	10	677	26.660	7480	1283	8840
6/2/70 - 8/3/71	Av	73.6	20.1	35.9	129.5	2.96	1723	16	906	29,018	10,378	2914	13,292
	Max.	79.4	22.3	37.2	136.4	4.12	3390	21	1097	32,300	12,996	5950	15,219
	Min	66.9	16.7	34.7	123.4	1.54	250	13	704	23,308	8500	1363	11,928

Table C-11

WHITE LIQUOR ANALYSES

Period		Sodium Compounds, gpl as Na_2O				Unreduced Na_2SO_4 as Na_2O, gpl	APHA Color, Units	Ca, ppm	Cl, ppm	COD, ppm	Carbon, ppm		
		Na_2CO_3	NaOH	Na_2S	Total						Inorganic	Organic	Total
6/15/69 - 8/11/69	Av	–	–	–	–	2.86	–	26	–	31,670	–	–	–
	Max.	–	–	–	–	4.64	–	64	–	40,832	–	–	–
	Min	–	–	–	–	2.02	–	8	–	25,120	–	–	–
11/3/69 - 12/16/69	Av	25.3	71.5	36.2	133.0	2.98	–	21	–	32,368	–	–	–
	Max.	31.0	76.3	40.9	135.1	5.56	–	29	–	34,860	–	–	–
	Min	20.5	62.8	32.9	130.4	1.52	–	16	–	29,700	–	–	–
1/20/70 - 1/26/70	Av	–	–	–	–	–	–	–	906	–	–	–	–
4/20/70 - 6/2/70	Av	22.9	75.0	35.4	133.5	3.26	–	18	951	30,814	2425	1311	3735
	Max.	26.7	78.1	37.2	138.3	4.52	–	30	1296	32,481	3420	1505	4788
	Min	19.2	72.5	33.5	130.2	2.18	–	15	675	25,294	1326	989	2881
6/2/70 - 8/3/71	Av	23.4	75.1	34.4	132.3	3.21	3045	26	964	28,927	3136	1094	4230
	Max.	26.6	78.1	37.8	135.8	5.04	5000	64	1339	34,980	3424	1700	4919
	Min	19.8	71.3	32.2	127.7	2.42	1500	11	895	22,972	2550	678	3674

Table C-12

TYPICAL OPERATOR'S DAILY REPORT

MASSIVE LIME SYSTEM FLOW RATES

Date: 2/21/71

Time	Bleach Effluent, gpm	Decker Effluent, gpm	Effluent To Slaker, gpm	Slaker Steam, 100 lb/hr	Lime Mud to Filter, gpm	Flue Gas (CO_2) cu ft/min	Lime Feed, Tons/Day	Slaker Temp, °F
7A	200	0	50	4	53	150	24.0	212
8								
9	200	0	50	4	58	150	24.0	212
10								
11	200	0	50	0	45	150	24.0	212
12N								
1	200	0	50	4	85	150	24.0	212
2								
3	200	0	50	2	85	150	24.0	212
4								
5	200	0	50	5	80	150	24.0	210
6								
7	200	0	50	0	70	150	24.0	212
8								
9	200	0	50	5	70	150	24.0	210
10								
11	200	0	50	5	35	150	24.0	212
12MN								
1	200	0	50	0	37	150	24.0	212
2								
3	200	0	50	8	32	150	24.0	212
4								
5	200	0	50	5	36	150	24.0	212
6								

Table C-13

TYPICAL OPERATOR'S DAILY REPORT
MASSIVE LIME SYSTEM OPERATING DATA AND ANALYSES

Date: 2/21/71

Time	Effluent Feed		Reaction Tank Titration, ml 1.0N HCL			Primary Effluent Clarifier Overflow — Titration, ml 0.1N HCL —				Carbonator Clarifier Overflow Titration, ml 0.1N HCL			Primary Clarifier Underflow, % Solids	Carbonator Clarifier Underflow, % Solids	Lime Mud to Filter, % Solids	Lime Mud from Filter, % Solids
	pH	Temp, °F	Pheno[1]	M O[2]	Temp °F	Pheno[1]	M O[2]	pH	Temp, °F	Pheno[1]	M O[2]	pH				
7 AM	9.0	134	9.2	9.5	165	15.4	17.4	12.4	161	9.2	13.2	11.4	19.2	21.2	19.2	50.6
8 AM																
9 AM	10.1	134	9.0	9.3	165	15.7	17.0	12.4	161	9.2	12.9	11.4	18.4	N.T.	20.4	52.0
10 AM																
11 AM	9.6	134	8.8	9.1	164	15.8	17.0	12.3	161	9.2	13.2	11.4	20.0	16.3	19.0	52.0
12 N																
1 PM	9.4	136	8.5	9.0	166	19.0	20.3	12.4	163	9.2	13.6	11.5	19.0	N.T.	18.0	50.0
2 PM																
3 PM	9.5	136	9.7	10.1	167	21.5	23.0	12.4	164	9.2	13.4	11.4	16.8	21.1	18.8	50.0
4 PM																
5 PM	9.1	132	8.6	8.8	164	20.0	21.5	12.5	161	9.2	13.7	11.5	16.4	N.T.	18.0	51.6
6 PM																
7 PM	9.6	134	8.0	8.3	163	18.5	20.0	12.5	159	9.0	13.0	11.5	17.2	18.4	18.0	52.4
8 PM																
9 PM	9.7	132	9.7	10.1	163	18.0	19.0	12.4	158	9.1	13.1	11.4	17.2	N.T.	18.5	52.0
10 PM																
11 PM	9.8	130	8.5	9.0	162	17.1	18.1	12.3	157	9.0	13.0	11.4	17.5	22.7	17.5	51.0
12 MN																
1 AM	9.6	128	7.8	8.2	161	15.8	17.0	12.4	156	9.3	13.3	11.3	18.0	N.T.	17.5	50.5
2 AM																
3 AM	9.4	126	9.7	10.1	160	18.5	20.0	12.4	155	10.0	13.8	11.4	16.0	18.3	17.0	50.0
4 AM																
5 AM	9.5	124	8.7	9.1	158	20.0	21.5	12.3	153	10.3	14.0	11.3	17.0	N.T.	17.5	51.5
6 AM																

[1] Titration to phenolphthalein end point.

[2] Titration to methyl orange end point.

Table C-14

TYPICAL OPERATOR'S DAILY REPORT FOR MASSIVE LIME SYSTEM PRIMARY CLARIFIER CONDITIONS AT SAMPLE POINTS[1]

	Percent Solids			Appearance		
Time	Sludge	Bottom	2nd	3rd	Top	Overflow
8:00 AM	19.2	12.0	0.4	Clear	Clear	Clear
12:00 Noon	20.0	12.0	1.2	Hazy	Clear	Clear
4:00 PM	16.8	8.4	5.6	Muddy	Very Cloudy	Hazy
8:00 PM	17.2	8.8	4.5	Muddy	Hazy	Clear
12:00 Midnite	17.5	9.3	4.0	Very Cloudy	Hazy	Clear
4:00 AM	16.0	10.0	3.6	Very Cloudy	Hazy	Hazy

COMMENTS: Backwashed filter at 7:05 AM. Backwashed filter at 2:15 PM. Increased mud flow from primary clarifier at 2:45 PM. Backwashed filter at 9:10 PM. Decreased mud flow to filter at 11:10 PM. Backwashed filter at 4:45 AM.

OPERATOR:

7:00 AM - 3:00 PM	3:00 PM - 11:00 PM	11:00 PM - 7:00 AM

[1] See Figure 4.

APPENDIX D

CAPITAL COST ESTIMATES

Table D-1

ESTIMATED CAPITAL COSTS FOR HYPOTHETICAL 1000-TONS-PER-DAY MILL

	Estimated Capital Costs, $		
	Without Massive Lime System	With Massive Lime System	Difference
Water treating and waste disposal	$ 4,387,000	$ 5,771,000	$ 1,384,000
Power plant	22,942,000	23,376,000	434,000
Pulp mill	29,814,000	29,997,000	183,000
Paper mill	26,827,000	26,827,000	—
Electrical facilities	950,000	950,000	—
Service facilities	2,247,000	2,247,000	—
Outside piping	1,290,000	1,320,000	30,000
Water supply and site facilities	5,399,000	5,490,000	91,000
Fire protection	300,000	300,000	—
Subtotal	$ 94,156,000	$ 96,278,000	$ 2,122,000
Sales and use tax	3,672,000	3,755,000	83,000
Contingencies	4,707,000	4,814,000	106,000
Engr., tools and temp. bldgs.	17,419,000	17,811,000	393,000
Total	$119,954,000	$122,658,000	$ 2,704,000

Table D-2

CAPITAL COSTS AFFECTED BY INCLUSION OF MASSIVE LIME COLOR REMOVAL PROCESS

	Estimated Capital Costs, $		
	Without Massive Lime System	With Massive Lime System	Difference
Water treating and waste disposal:			
Primary waste treating equipment	$ 760,000	$ 715,000	$ (45,000)
Secondary waste treating system	1,550,000	1,535,000	(15,000)
Color reduction system	--	1,444,000	1,444,000
Power plant:			
Turbine generator	3,470,000	3,566,000	95,000
Recovery boiler	9,325,000	9,500,000	175,000
Turbine and evaporator condenser cooling tower	300,000	310,000	10,000
Black liquor evaporator equipment	2,111,000	2,234,000	123,000
Black liquor oxidation equipment	310,000	328,000	18,000
Desoaping equipment	202,000	214,000	12,000
Pulp mill:			
Brown stock washers	2,356,000	2,358,000	2,000
Caustic plant equipment	1,489,000	1,609,000	120,000
Lime recovery equipment	2,230,000	2,291,000	61,000
Outside piping:			
Pipe, valves and fittings for			
Process	151,000	161,000	10,000
Steam and condensate	66,000	70,000	4,000
Oil piping	57,000	60,000	3,000
Labor	420,000	433,000	13,000
Water supply and site facilities:			
Railroad facilities	387,000	401,000	14,000
Process sewers	211,000	228,000	17,000
Piling	1,389,000	1,449,000	60,000
Subtotal	$26,784,000	$28,906,000	$ 2,122,000
Sales and use tax			83,000
Contingencies			106,000
Engineering, tools and temporary buildings			393,000
Total			$ 2,704,000

*U.S. GOVERNMENT PRINTING OFFICE :1973 514-193/195 1-3